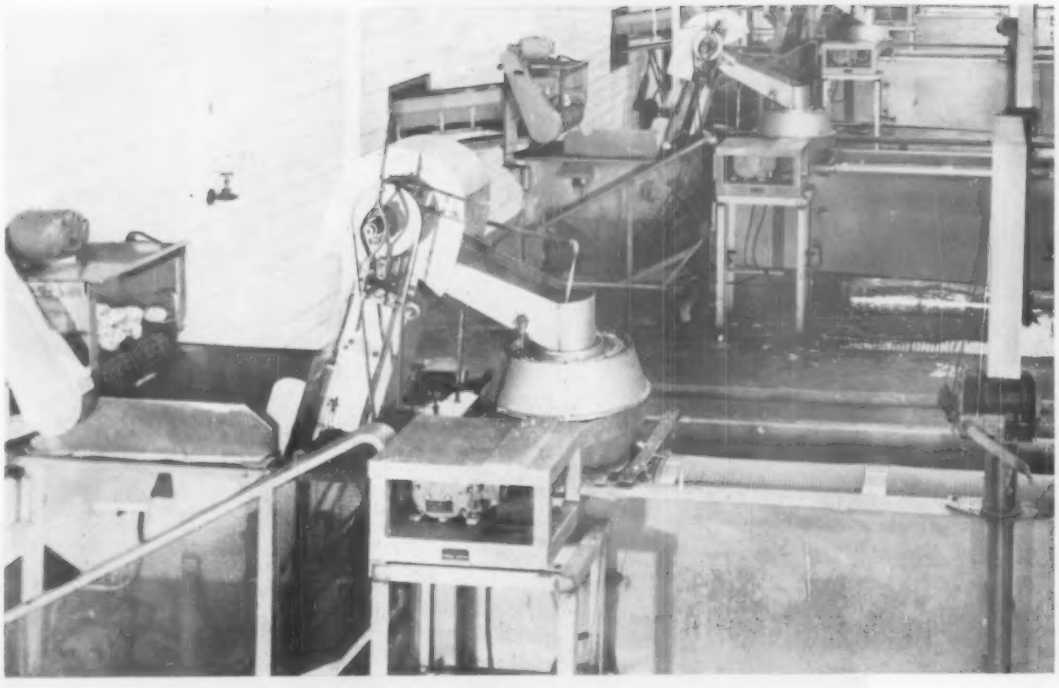
SEPTEMBER, 1960
Vol. 14, No. 9

FT food technology



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Economic Trends in Food Processing in the South[a, b]

N. M. Penny
Georgia Experiment Station,
Experiment, Georgia

AN AMAZING food processing industry, which in many respects has made possible both today's type of agriculture and today's processed, packaged, and serviced food supply, stands between the farmer at one end of a continuous flow of supplies and the retail store at the other.

Economic, social, and technical developments in the Nation and in the South made possible and necessary the growth of the food processing industry. Increases in real per capita incomes, shifts in population from rural to urban living and, consequently, to different patterns of social life, and the rapid advances in all phases of science and technology—these are the principal factors that opened the way for a highly technical, specialized, commercialized food processing industry. These social and economic changes and the emergence of the modern food processing industry appeared in other regions of the Nation sooner than in the South.

For a long period prior to the second World War, agriculture in the South had undergone little change either as a type of farming or as a way of life. Agriculture was generally characterized by a large number of relatively small farms producing a diversity of crops, an abundance of farm family labor, and subsistence farming. Subsistence farming varied with the degree to which specialization and commercialization had advanced in a locality and to some extent with the size of farm. The usual family farm produced much of its poultry, meat, vegetables, and fruit for home consumption. These foods were eaten fresh in season and in preserved form during the off-season.

Since World War II the surge in science and technology has shown its effects on southern agriculture. The growth of urban centers, paralleling the increase in industry and commerce, has provided off-farm employment opportunities and the population has drifted from rural to urban centers. The size of farms is increasing and capital assets are being substituted for the labor asset. As a result, output per man and real income are increasing. Farming continues to move in the direction of specialization and commercialization. Subsistence farming continues to decline. The effect of all this has been that the farmer who moves to the city becomes wholly dependent on the food industry for food supplies, and the farmer becomes almost as dependent.

Consumers in recent years have steadily increased their dollar outlays for food as real incomes have increased. The reduction in home production, an increased trend toward eating meals away from home, and more marketing services have also contributed to this increase in per capita food expenditures.

Moreover, consumption patterns have been changing in the South. Consumption of pork and lard and corn meal has decreased. The shift has been toward the use of more processed fruits and vegetables and livestock products and away from cereals and potatoes. Consumption of potatoes and processed fruits and vegetables was relatively low in the South and relatively high for fresh vegetables. The trends toward eating more meals away from home, buying more prepared meals for home use, and purchasing more food services and conveniences seem certain to continue to increase. The food processing industry is developing in the South in response to these discernible transformations in its economic, social, and technologic structure. The South is presently advancing, in fact, at a faster rate than other regions of the country.

The accompanying charts show graphically the rapid growth and development of food processing in 15 southern states from Maryland to Texas including

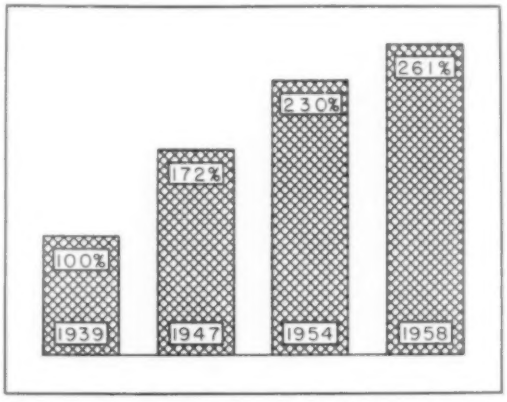

Figure 1. Growth of food manufacturing in the South in terms of constant dollars (1947-49 price level).

SEPTEMBER COVER. Birth of a potato chip! Depicted is a series of Urschel Model "CC" slicers developed for the potato chip industry, and installed in Jays Foods Inc. Potato Chip Plant, Chicago, Illinois. Potatoes emerge from the left and are dropped into a hopper where separation of gross foreign material is accomplished. They are then dropped into the slicer proper where centrifugal force causes potatoes to press against gauging shoes with approximately seven times their weight. While influenced by this force they slide over a series of eight knives (disposable) in the slicing head where both slicing and separation of fine foreign matter occurs. Potato slices are then discharged against a conical ring, dropped through the discharge chute, and conveyed for further processing.

[a] Presented at the Better Foods for Better Nutrition Conference, University of Georgia, Athens, Georgia, March 3, 1960.

[b] Acknowledgments are made to Mr. K. E. Ford, Associate Economist, Georgia Experiment Station, for help in the preparation of this publication.

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FT food technology

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the District of Columbia. They provide a comparison with the United States as a whole.

The percentage change from 1939 in terms of constant dollars, using the 1947–49 price level for deflating the dollar, is shown in Figure 1. Put in terms of the 1947–49 dollar, food processing in the South as of 1958 had increased 161% from what it was in 1939. In actual dollars the value of foods processed in the South was 1 billion 690 million dollars in 1939, 11 billion 312 million in 1958!

In the same period food processing in the United States as a whole increased in terms of the same constant dollar by 93% (Figure 2). The total value of foods processed in the United States was 10 billion 618 million dollars in 1939 and 52 billion 581 million dollars in 1958. That total food processing in the South in 1958 exceeded total food processing in the United States in 1939 is rather amazing.

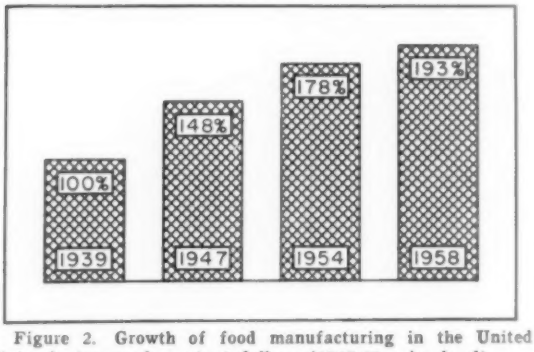

Figure 2. Growth of food manufacturing in the United States in terms of constant dollars (1947-49 price level).

Growth of food manufacturing in one southern state (Georgia) is compared graphically in Figure 3 with that in the South and in the United States. Since 1939 Georgia has exceeded the South as a region and the United States as a whole in rate of growth in food manufacturing. These comparisons are in terms of the deflated or constant dollar. Much of the growth in Georgia is clearly attributable to the broiler processing business.

In Figure 4 some other southern states are included to show the relative changes in states that were, among the southern states, low in actual dollar value of food manufacturing in 1939 and have made less percentage change. For example, the output of South Carolina's food processing industry had only a 31 million dollar value in 1939 but increased it by 1958 to 174 million or 118% in terms of deflated dollars. Alabama, with a total value of 52 million dollars in 1939, increased that value to 454 million in 1958—241% in terms of constant dollars. In another state, Texas, the value was fairly high in 1939, 341 million, but rose to 2 billion 351 million in 1958, a 169% increase in constant dollars.

Some facts not shown in charts are of considerable interest. The total number of food manufacturing establishments decreased in the South and in the United States between 1947 and 1954 but within some classes of food manufacture there was an increase.

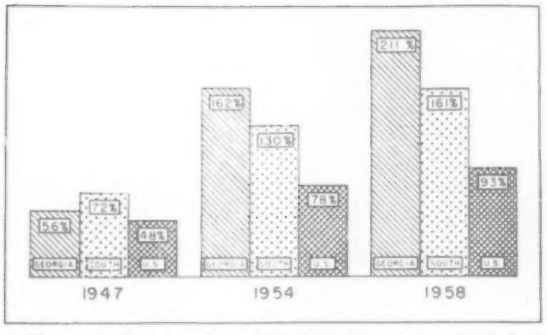

Figure 3. Increase from 1939 in value of output of food manufacturing: Georgia, the South, and the United States as a whole in terms of constant dollars (1947-49 price level).

For example, though dairy plants, canning, preserving and freezing plants, and miscellaneous food preparation enterprises declined in number, meat processing plants increased both in the South and in the United States as a whole. It is of interest that grain-mill products plants increased in the South but decreased considerably in other regions of the United States.

The number of employees rose sharply in the meat processing business in the United States and the South but declined in number in the United States and South in dairy products and miscellaneous food preparation plants. Changes in number of employees were relatively small up or down in canning, preserving, and freezing, and in the grain-mill products branch of the food industry.

It can be stated with assurance, in concluding this brief assay of economic trends in food processing in the South, that the gains made in the southern states have been substantial. Measured by the constant dollar yardstick, the distance covered between 1939 and 1958 is impressive, one of the signal events in the U. S. economy of that span of years.

Figure 4. Increase in value of output of food manufacturing from 1939 to 1958, selected states compared with the South and the United States at large in terms of constant dollars (1947–49 price level).

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Committee for the 1961 Technical Program Appointed

President Imri Hutchings, acting on the recommendations of the Council, has appointed a permanent program committee having a rotating membership. Five members will be replaced each year—one in each section. The complete membership for 1961 follows:

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FUTURE MEETINGS FOR FOOD TECHNOLOGISTS

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September 11-16	American Chemical Society's 138th national meeting, New York, New York
September 20-21	Military-Industry Conference on Freeze-Drying and other Dehydration Methods of Food Preservation, Sponsored by Research and Development Associates, Stock Yard Inn, Chicago, Illinois. (For information write Col. R. A. Isker, U.S.A. ret'd, Research and Development Associates, 1819 W. Pershing Road, Chicago, Ill.)
September 20-23	Pure Food Centenary, Royal Institution of Great Britain, London, England
October 4-7	Tenth Annual Instrument Symposium and Research Equipment Exhibit, National Institutes of Health, Bethesda, Maryland
October 12-14	The Seventh National Symposium of the American Vacuum Society, Cleveland-Sheraton Hotel, Cleveland, Ohio
November 12-19	Fifth Pan-American Congress of Pharmacy and Biochemistry, Santiago, Chile. (For information address Senor José Pérez Molina, Secretary, Box 6084, Santiago, Chile).

1961	
May 7-11	**Twenty-first Annual Meeting of the Institute of Food Technologists, Hotel Statler, New York, New York**
1962	
June 10–14	**Twenty-second Annual Meeting of the Institute of Food Technologists, Fontainebleau Hotel, Eden Roc Hotel, Miami Beach, Florida**
1963	
May 19–23	**Twenty-third Annual Meeting of the Institute of Food Technologists, Pantlind Hotel, Civic Auditorium, Grand Rapids, Michigan**

An open invitation is extended to readers of FOOD TECHNOLOGY to send in to the Editorial Office, 11606 South Bell Avenue, Chicago 43, Illinois notices of annual or national meetings of interest to food technologists.

REGIONAL NEWS

INDIANA

The Indiana Section will serve as host to the Chicago Section Saturday, October 15, 1960, date of the Ohio State University-Purdue football game for the first meeting of the 1960–61 year.

The following schedule has been planned:

Tour—10:45 a.m. (CDT). Meeting Place—Purdue Dairy Center.

Food—11:30–12:30 p.m. Barbecue chicken luncheon at the Dairy Center.

Game—1:30 p.m. O. S. U. (vs) Purdue at Ross Ade Stadium.

A special executive meeting was held at Muncie at the summer cottage of Chairman Fred H. Dellwo on June 3, 1960. An evening of fellowship was enjoyed by all. The evening was also one of business, covering organization and planning for the 1960–61 year. The following list of members will comprise the executive committee of the section for the 1960–61 year: F. H. Dellwo, Chairman; G. M. Clark, Chairman-Elect; William J. Klinker, Secretary-Treasurer; L. F. Rutledge, Councilor; N. R. Tarvin, Chairman ex-officio; J. Unger, R. E. Noble and V. Walter.

IN THIS ISSUE–

TECHNICAL ARTICLES IN BRIEF

Built-in defrost indicator — *Psychrophilic bacteria*

Role of psychrophilic bacteria in frozen food spoilage. A. C. Peterson and M. F. Gunderson.

Psychrophilic bacteria in frozen foods stored at defrost temperatures were studied. The liberation of potent extracellular proteolytic enzymes in the growth menstruum at times and temperatures which migh be expected to afford the product some protection was demonstrated. Development of off-flavors and odors in defrosted chicken pies in advance of the development of great numbers of psychrophilic bacteria was noted. A psychrophilic culture of *Pseudomonas fluorescens* with marked enzymatic activities was isolated. The proteolytic and saccharolytic activities of both extra- and endocellular enzymes from this microbe was demonstrated. Previously observed defrost effects were duplicated by the addition of these enzymes to normal chicken pies. It was demonstrated that once the enzymes of psychrophilic bacteria are elaborated and liberated in the growth menstruum, lowering the temperature will slow their action, but not stop it. Definitely it will not remedy damage already done. Therefore, frozen foods can be said to have a built-in indicator of defrost. This indicator is the effect of psychrophilic bacteria and their enzymes on the physical characteristics of the product. (See page 413).

Evaluation — *Effect of different fats in the feed on egg quality*

Functional properties and flavor of eggs laid by hens on diets containing different fats. Ruth Jordan, Gladys E. Vail, J. C. Rogler and W. J. Stadelman.

Eggs from three groups of Leghorn hens on diets containing different kinds of fat were compared with respect to stability of whole-egg foams as measured by percentage of drainage in a one-hour period, leavening power as indicated by volumes of whole-egg sponge cakes, and flavor. Diets for the 3 groups of hens were, respectively, a stock ration supplemented with 10% corn oil, the same stock ration supplemented with 10% beef tallow, and the stock ration only. During the period that the 3 groups of hens were fed the same diet, no significant differences were found among foams from eggs of the 3 groups. After hens were on the experimental diets for 3 weeks or longer, percentage of drainage from the foams of the beef tallow group eggs was significantly greater than from eggs of the corn oil group; after 6 weeks, foams from both beef tallow and stock ration groups had higher percentages of drainage than those from the corn oil group. Whole-egg sponge cakes made from eggs of the beef tallow group were significantly smaller than similar cakes made from the other 2 groups of eggs. Flavor tests on freshly laid eggs showed distinguishable differences among the 3 groups; however, after 3 months' storage under refrigeration, no significant difference was found between eggs of the corn oil and stock ration groups; a highly significant difference in flavor between the beef tallow and the stock ration groups was found. Products made from eggs of the corn oil group were noticeably lighter in color than similar products from the other 2 groups of eggs. Inasmuch as iodine values of the ether extracts of egg yolks from the 3 groups of hens were found to differ significantly, a possible relationship between the nature of egg yolk fat and functional properties of the egg was suggested. (See page 418).

Determination of soluble solids — *Citrus juices*

The determination of soluble solids in citrus juices. I. The effect of non-sucrose components on refractometer values. W. Clifford Scott, Donald A. Morgan, and M. K. Veldhuis.

Effects of non-sucrose components of citrus juices on their refractometer sugar scale values were studied. The only two components whose refractive indices in solution have been compared with sucrose and reported in the literature are citric acid and reducing sugars. Both have lower indices of refraction than sucrose, and therefore require positive corrections for the refractometer sugar scale to indicate true soluble solids. Other components studied had higher indices of refraction than sucrose. Negative corrections required for both amino acids and pulp are greater than positive corrections required for citric acid at equivalent concentrations. Negative corrections required for citrate salts, flavonoids, and pectin are considerably less; those for ascorbic acid and peel oil are practically negligible. (See page 423).

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Analytic method — *Constituents of beef*

Determination of the proximate and electrolyte content of beef. Morris D. Berman.

A convenient plan of analysis for the proximate and major electrolyte content of beef is described. With this procedure, the coefficient of variation for the constituents was as follows: moisture, 1.21%; ash, 2.94%; protein, 0.69%; fat, 5.62%; chloride, 2.89%; magnesium, 2.05%; calcium, 0.68%; zinc, 3.15%; sodium, 4.21%; phosphorus, 0.92%; potassium, 1.18%; and iron, 2.26%. Average recovery of various ions was: chloride, 106%; magnesium, 103%; calcium, 97%; zinc, 94%; sodium, 97%; phosphorus, 99%; potassium, 103%; and iron, 102%. (See page 429).

Additives — *Effect of sodium chloride, citric acid, sucrose on canned tomatoes*

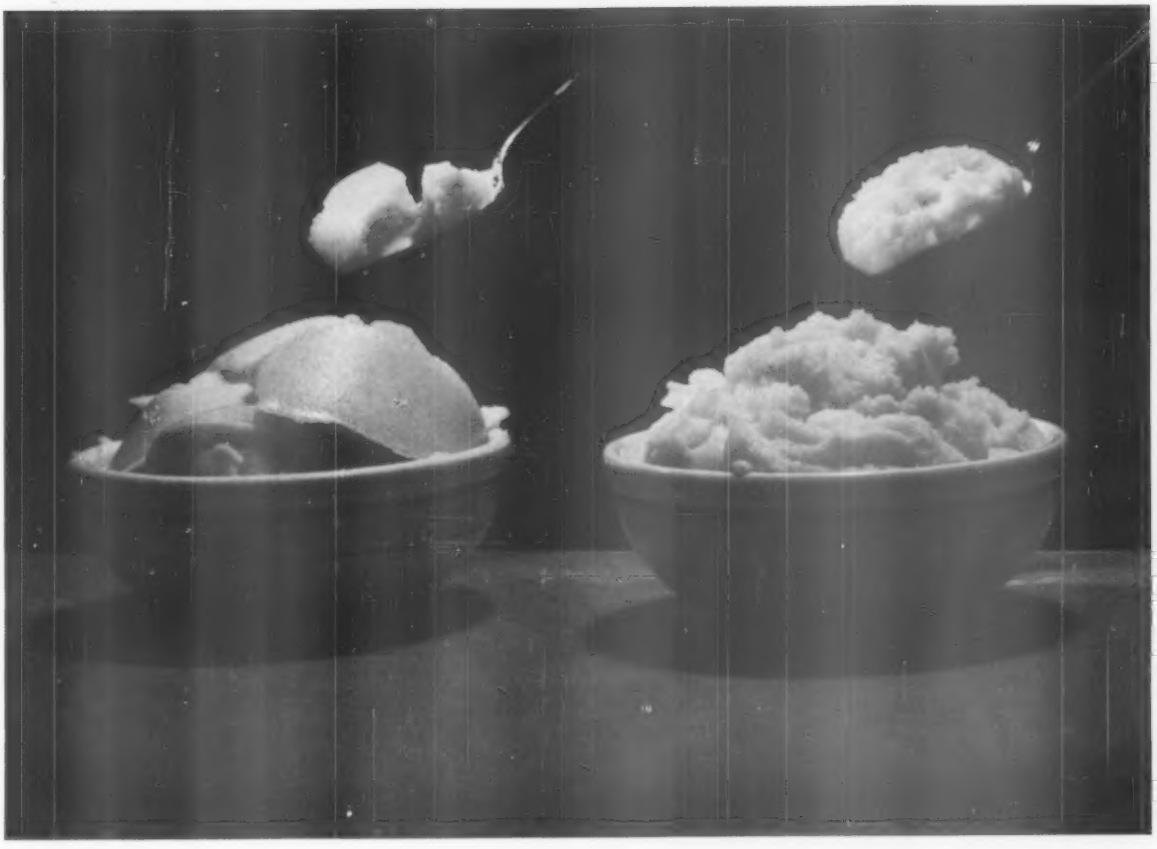
Effect of sodium chloride, citric acid, and sucrose on pH and palatability of canned tomatoes. Sherman Leonard, B. S. Luh, and Rose Marie Pangborn.

To selected lots of California tomatoes, acidified with citric acid to lower the pH and to facilitate heat sterilization, varying amounts of sodium chloride and sucrose were added. Tomatoes were of 5 varieties. Three months after canning, the tomatoes were analyzed for pH, acidity, soluble solids, drained weight, and flavor acceptability. Adding citric acid effectively lowered pH but impaired palatability. The proper combination of citric acid, sucrose, and sodium chloride improved flavor quality over untreated controls within each variety. Agtron E color readings decreased from 66 in under-ripe to 42 in canning-ripe, and to 35 in soft-ripe tomatoes. Average pH values increased from 4.33 in under-ripe to 4.48 in canning-ripe, and to 4.64 in soft-ripe fruit. Changes in pH during storage of the canned product for 3 months at room temperature are discussed. (See page 433).

Firming agent — *Calcium hydroxide added to canned peppers*

Use of calcium hydroxide for firming canned green and red sweet bell pepper. Maurice W. Hoover.

The effect of calcium hydroxide and other calcium compounds on the firmness of canned pepper was determined. Among the compounds evaluated were calcium hydroxide, calcium chloride and calcium sulfate. Effects of various methods of treating, duration of treatments and calcium concentrations on the firmness of canned pepper were evaluated. Influence of storage on the firmness of canned pepper was determined. Calcium hydroxide was very effective as a firming agent for canned pepper when applied to fresh pepper prior to heat processing and was the most effective firming agent evaluated. Good results were obtained by all three of the methods used to apply the lime treatments. Best results were obtained in cubed pepper when the lime was applied at the rate of 0.2 to 0.4%, by weight, for approximately 30 minutes followed by a thorough rinse to remove the lime residue prior to blanching and heat processing. When pepper halves were treated, it was necessary to extend the treatment time to 2.5 hours or longer for optimum results. Green pepper responded better to firming treatments than red pepper. (See page 437).



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C. Description of Product or Process.
D. Statement of reason for considering this a meritorious achievement.
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Germicidal effect — *Orange peel oil and D-limonene*

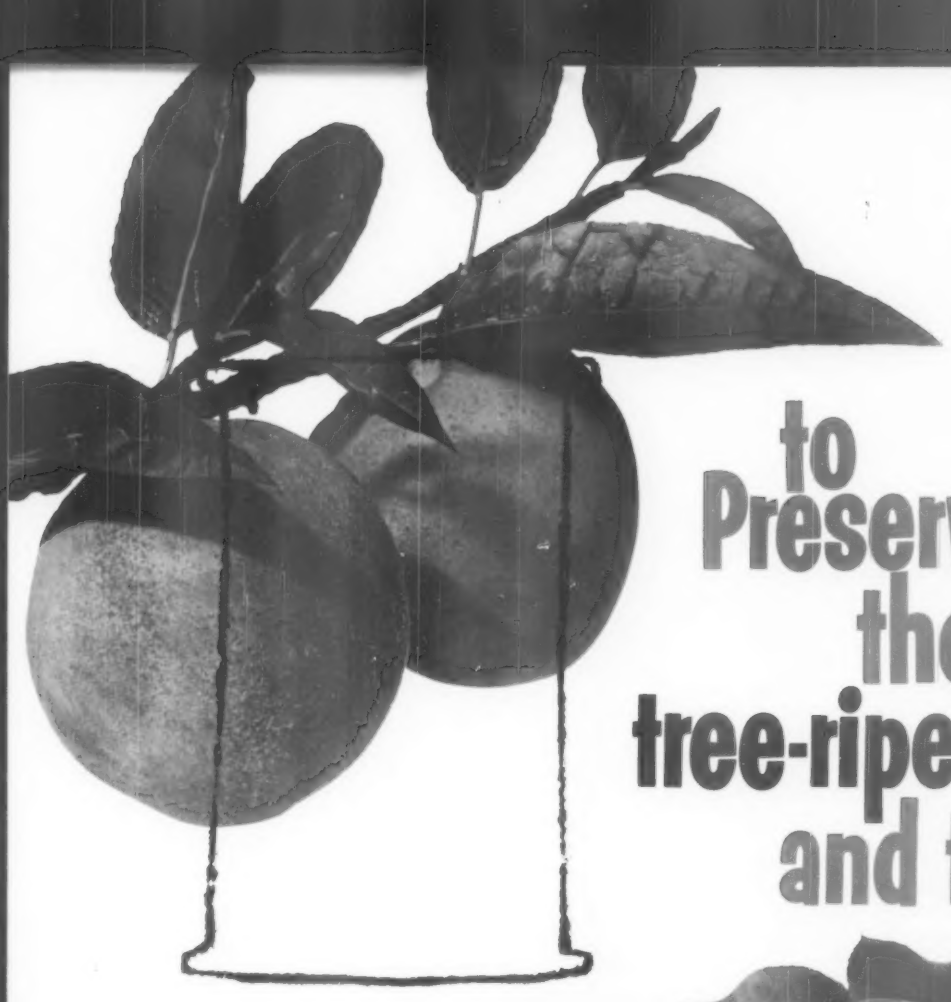
Germicidal effect of orange peel oil and D-limonene in water and orange juice. I. Fungicidal properties against yeast. D. I. Murdock and W. E. Allen.

Fungicidal effect of orange peel oil and D-limonene (stripper oil) was determined against *Zygosaccharomyces major* in water and in single strength orange juice at 25° C. Both oils exhibited lethal properties in water (pH 7.0) in concentrations as low as 0.02%; in orange juice 0.1% was required. D-limonene was more effective than orange peel oil in both water and orange juice. Fungicidal properties of these oils in orange juice adjusted to various pH levels were determined. Orange peel oil and D-limonene were more effective at pH 6.0 and 7.0 than they were at pH 3.0 and 4.0. Orange juice adjusted to pH 7.0 with NaOH and containing 0.1% D-limonene was sterile 3 hours after being inoculated with 1,000,000 yeast cells per ml. In juice adjusted to pH 3.0 with citric acid, there were 100,000 viable cells per ml remaining at the end of a like period. Results indicate that the preservative properties of sodium benzoate can be greatly enhanced by adding orange peel oil and D-limonene in concentrations as low as 0.02%. It appears that these oils are synergistic to sodium benzoate. (See page 441).

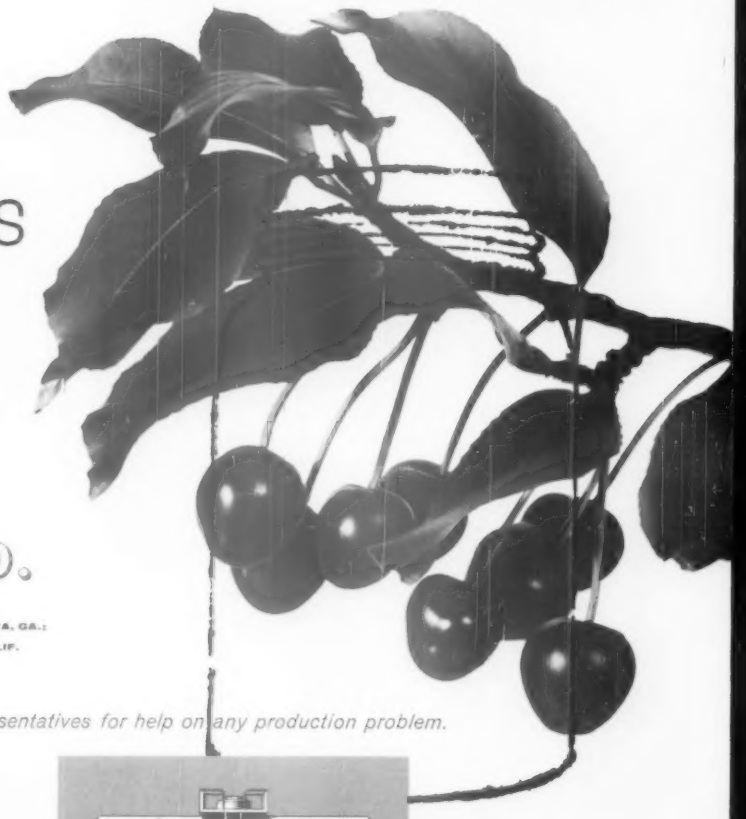
Quality study — *Effect of partial pumping of hams*

Effects of partial pumping on the shrinkage, rancidity development, and palatability of aged hams. George C. Skelley, Jr., James D. Kemp and W. Y. Yarney.

Sixty-four hams were divided into 8 equal groups. Four groups were dry-cured, and the companion hams in 4



to Preserve the tree-ripe flavor and texture...



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groups were partially pumped with an 85° pickle made from the dry-cure mixture to 4, 6, 8, and 10% respectively, and were then rubbed with dry-cure mixture to allow the total curing ingredients to equal the dry-cured groups. All hams were cured for 21 days at 36–40° F (2.2–4.4° C). After smoking at a smokehouse temperature of 100° F (35.6° C), the hams were aged for 6 months at a temperature of 65° F (18.3° C) and a relative humidity of 55%. Hams were weighed to the nearest 0.01 pound when fresh, after pumping, after curing, after smoking, and at monthly intervals for the 6 months aging period. Fat characteristics of external fat were determined by iodine numbers, peroxide numbers, and free fatty acid content. After 6 months' aging, fatty acid content and peroxide numbers were determined for seam fat. Hams were cut and observed for color, soundness and odor. Two slices were broiled for palatability study, and a third was analyzed for sodium chloride. Shrinkage increased throughout processing and aging, with the greatest amount occurring during the first month of aging. Pumping reduced shrinkage throughout aging. Iodine numbers were not affected by curing method. Peroxide numbers and free fatty acid content increased during aging with no significant difference in external fat due to method of curing. Free fatty acid content was higher, peroxide numbers were lower in seam fat of pumped hams. Salt content was higher in pumped hams with a general increase as pumping rate increased. With the exception of hams pumped 4%, dry-cured hams were sounder, showed a more desirable color, and were scored significantly higher by a palatability panel for saltiness, flavor and over-all satisfaction with no difference in tenderness. Hams pumped 4% were quite similar in quality to dry-cured hams. (See page 446).

Processing methodology — *Infrared used as blanching agent*

Studies on the application of infrared in food processing. E. A. Asselbergs, W. P. Mohr, and J. G. Kemp.

Three types of high energy, infrared radiators—quartz lamps, quartz tubes, and calrods—were evaluated for their effectiveness in blanching apple tissue. Data are presented which show how the depth of heat penetration was influenced by wavelength characteristics, voltage input, and energy output of the radiator. Infrared radiation was used successfully for blanching celery, apples, and peas prior to freezing and in the preparation of french fried potatoes. General quality of infrared-blanched samples was better than that of samples blanched by conventional steam or boiling water methods. Accompanying weight losses of up to 35% by weight would result in reduced freezing, storing, and handling costs in the case of products intended for later remanufacture. Infrared was substituted for the par-boiling treatment of beef as preliminary heat treatment in preparing beef stew. The color, texture, and flavor of the infrared-braised, canned meat was considered superior to that of par-boiled meat. (See page 449).

Engineering analysis — *Microwave vs conventional electric oven*

Comparative heating efficiencies of a microwave and a conventional electric oven. George A. Pollak and Louise C. Foin.

The heating efficiency of microwave and conventional cooking equipment in the heating of water and the roasting of

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beef was studied. In heating water, the over-all electrical efficiency was 34% for both microwave and conventional ovens, but the efficiency of a conventional hot plate in heating water was 63%, virtually double that of the ovens. In roasting 8-lb ribs of beef, the over-all electrical efficiencies were 36.7% for the conventional and 33.4% for the microwave oven. However, the product prepared in the conventional oven yielded 13% more edible meat using 24% less electrical energy. A better way of expressing the energy-yield relation is the energy required to prepare a unit weight of edible product. This was found to be *74 BTU per ounce of yield* for the conventional as compared to *119 BTU per ounce of yield* for the microwave oven. The difference in energy was attributed primarily to energy required to evaporate a greater amount of water in microwave cooking. (See page 454).

Cooking rate — *Effect on fresh-unfrozen and on fresh-frozen turkey meat*

Comparison of rate of cooking and doneness of fresh-unfrozen and frozen, defrosted turkey hens. Graycee E. Goertz, Anna S. Hooper, and Dorothy L. Harrison.

Fresh-unfrozen and fresh-frozen turkeys were roasted to 90° C in the breast and to 95° C in the thigh. Turkeys roasted to 95° C in the thigh generally were more done than those roasted to 90° C in the breast. Total cooking time was longer ($P<.05$) for fresh-frozen turkeys roasted to 95° C in the thigh than for fresh-frozen turkeys roasted to 90° C in the breast. Similar results for cooking time were noted for fresh-unfrozen turkeys. Also, fresh-unfrozen and fresh-frozen turkeys and turkeys stored 1 and 3 months at 0° F were roasted to 95° C in the thigh. Total cooking time was shorter ($P<.05$) and volatile cooking losses lower ($P<.05$) for fresh-unfrozen turkeys than for the frozen turkeys. Dripping losses were lower ($P<.05$) and light meat flavor scores and dark meat tenderness scores higher ($P<.05$) for turkeys stored 1 and 3 months than for the fresh-unfrozen or fresh-frozen turkeys. Doneness scores for light and dark meat were higher ($P<.05$) for turkeys stored 1 month than for those of the other turkeys; whereas doneness scores for uncarved turkeys generally were similar. In addition, frozen, defrosted turkeys were roasted breast-up or breast-down. The thigh temperature of turkeys roasted breast-up increased more rapidly and cooking time was shorter ($P<.05$) for turkeys roasted breast-up than for those roasted breast-down. Doneness and other palatability scores generally were similar for all birds in this group. (See page 458).

Maturity indices — *R. I. Greenings and Baldwin apples*

The relationship of apple maturity to applesauce quality. R. L. LaBelle, R. S. Shallenberger, R. D. Way, L. R. Mattick, and J. C. Moyer.

Maturity indices of R. I. Greenings and Baldwin apples (1957 and 1958 harvests) were evaluated. Their relationship to the quality of sauce processed from these apples both with and without storage ripening was also investigated. An applesauce pilot plant of 300 lb per hour capacity was used to process sauce from this fruit under precisely controlled conditions, the equipment closely simulating commercial processing. Pressure test was discarded as a prime index of maturity at harvest in favor of soluble

(Continued on page 22 following Technical Section)

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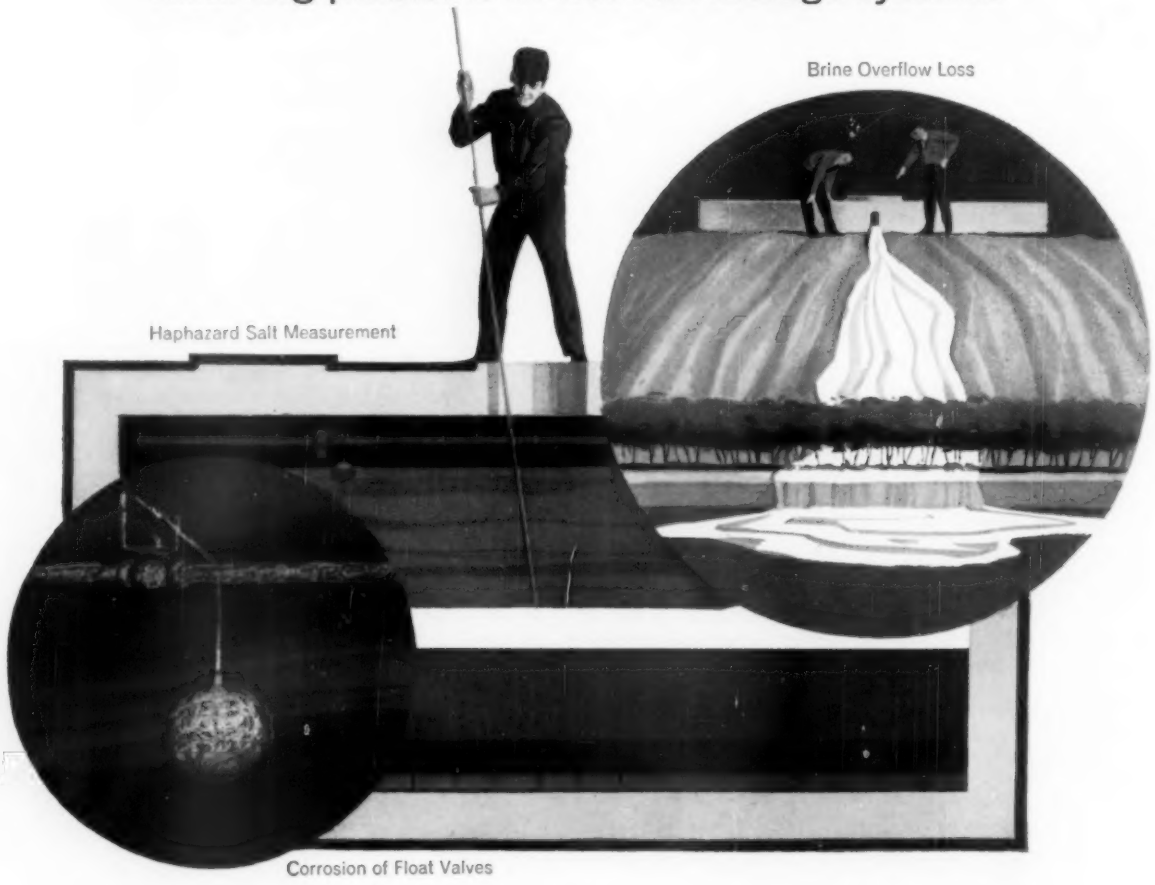

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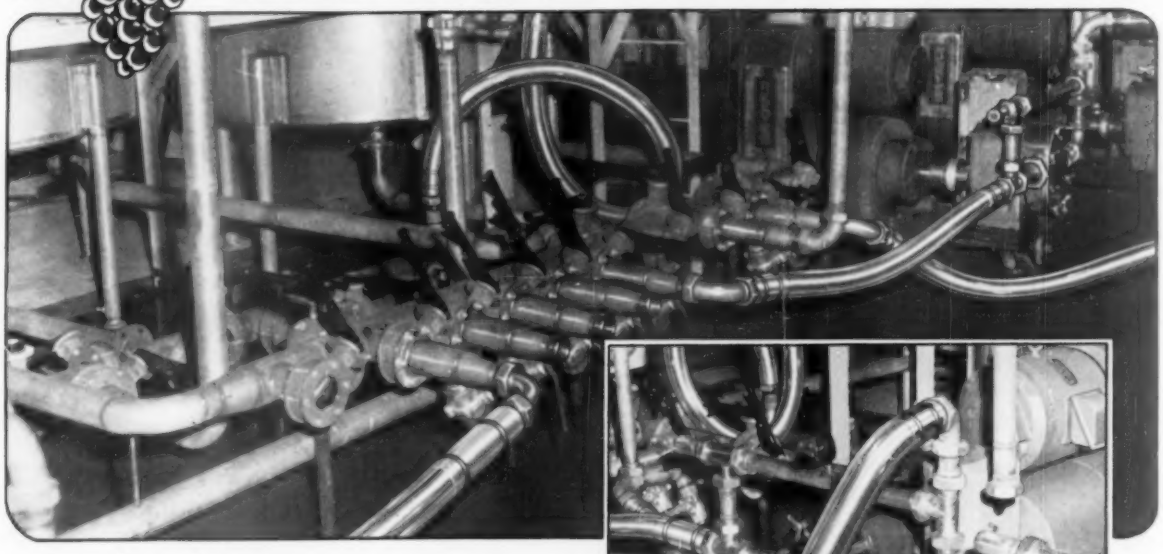

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Role of Psychrophilic Bacteria in Frozen Food Spoilage

A. C. Peterson and M. F. Gunderson

Department of Bacteriological Research, Campbell Soup Company, Camden, New Jersey

(Manuscript received March 3, 1960)

OBSERVATIONS of defrosted frozen foods and products kept at temperatures slightly below 32° F showed that the damage suffered by the products was far out of proportion to that which might be ascribed to the bacterial populations developed at these defrost temperatures. It was postulated that the enzymes of bacteria which grow at temperatures below 41° F were responsible for the damage; namely, the extensive changes in the physical and organoleptic characteristics of the foods, including rancidity, destruction of the colloidal character of the gravies of chicken pies and similar products, and pronounced flavor and aroma changes. Although temperatures considered adequate for the storage of frozen foods and for maintaining them in a hard frozen condition, have progressively changed from 20° F to 10° F and down to 0° F, this work and the continuing activity in this field are a recognition of the problems presented by bacteria growing at or near the defrost temperature of 32° F. This study also emphasizes the necessity of keeping the growth of psychrophilic bacteria in check and takes cognizance of the possibility of enzymatic and nonenzymatic chemical changes not directly dependent upon active bacterial multiplication.

Psychrophilic bacteria, those bacteria growing well at refrigerator temperatures, are known to have adverse effects on the storage life and quality of many food products, particularly dairy products *(9)*. These bacteria are responsible for many undesirable changes —unclean, sour odors and tastes, putrid and fruity odors, ropiness and surface discoloration and general impairment of keeping quality.

United States Department of Agriculture workers *(1)* have pointed out that enzyzme reactions are going on at all temperatures. Cold slows them down, but does not stop them. Lineweaver *(8)* has shown that enzyme catalyzed reactions with low temperature coefficients (Q_{10}) become relatively greater in importance than both organized physiological processes and nonenzymatic chemical reactions as the temperature is lowered. Balls and Lineweaver *(3)* pointed out that proteolytic activity proceeded in the solid state, but that such activity was limited by lack of fluid, which prevented diffusion and thus prevented new contacts between enzyme and substrate. They suggested that where the substrate was present in large masses, the diffusion effects were less, and enzymes would probably show much more activity in the frozen state. Balls and Lineweaver have shown this to be particularly true with insoluble protein fibers where proteolysis proceeds along a fiber as contrasted with soluble proteins where contact between enzyme and substrate are limited by the solid state. Lipase action of psychrophilic microorganisms is apparently greater than that of the proteinases and proceeds at low temperatures and in the frozen state. The lower fats and unsaturated fats are not well protected from enzyme action by lowering the temperature. Weiser *(12)*, however, has stated that microbial enzyme activity is appreciably affected by low temperatures.

Prescott and Tanner *(10)* have noted the selective action of low temperatures (between 32° F and 41° F) on the ordinary organisms of spoilage and on saprophytes in general. The found that organisms which attacked proteins did develop at those temperatures, whereas carbohydrate fermenters were suppressed. A study of microorganisms growing on frozen meats *(11)* showed that the majority of organisms were pseudomonads that did not exactly fit Bergey's classification. Similar results were reported by Berry *(4)* in a study of frozen fruits and vegetables.

The present study was undertaken to investigate the possibility that enzymes from psychrophilic bacteria are involved in the spoilage of frozen foods at defrost temperatures.

EXPERIMENTAL METHODS

Chicken pies and turkey gravy were obtained from production sources. The experiments were performed using a number of pies since none of the pies contained enough gravy for repeated sampling. Consecutive pies from the same gravy batch were obtained for these experiments. By using consecutive pies, it was believed that variations from one pie to another were minimized. The pies were baked and examined organoleptically at the same time they were sampled bacteriologically.

Simple 1% starch gels were prepared using distilled water and corn starch which were then autoclaved to ensure sterility.

Water extracts of the gravies of defrosted chicken pies were prepared by removing as much of the liquid and gravy contents of the pie as possible to a tared sterile empty dilution bottle. Care was taken to avoid as much meat and vegetable solids as possible. An equal weight of sterile water was added and the mixture shaken vigorously for 3 min. The sample was then centrifuged at 33,000 x g in a Sorvall[a] Super Speed SS-1 Centrifuge for 10 min. The supernatant fluid was pipetted off and passed through a Selas filter.[b] These samples were frozen until used.

Proteolytic activity was determined by the method of Anson *(2)*. After reaction of the proteolytic enzymes with the casein test substrate, the amount of tyrosine, tryptophane and peptides containing these two amino acids in positions where they can react with Folin-Ciocalteau reagent, in the trichloroacetic acid soluble fractions was determined. After reaction with the phenol reagent, the intensity of light transmitted at 645 mme was measured.

The culture of *Pseudomonas fluorescens* used in these experiments was obtained from a defrosted chicken pie stored at

[a] Ivan Sorvall, Inc., Norwalk, Conn.

[b] Manufactured by Selas Corporation of America, Dresher, Pa.

41° F for 5 days. The culture was isolated from a tryptone-glucose-meat extract agar plate (TGE) after incubation for 7 days at 41° F. Tryptone-glucose-meat extract agar and broth of pH 7.1 were used for the cultivation of this organism. Generally, the culture was grown at 41° F for 5 days. The bacterial cells were recovered from the agar slants by washing off with 0.02 molar Sorensen's phosphate buffer *(5)* of pH 7.4 and centrifuging the cell suspension. Similarly, the bacterial cells were recovered from broth by centrifugation. The cells were washed 5 times in phosphate buffer and the packed volume determined at 1500 rpm (600 x g) for 30 min in an International size 2, model V centrifuge. The packed cells were diluted 1 to 4 by volume in the same buffer.

Extracellular proteolytic enzymes were recovered by precipitation at 85% saturation with ammonium sulfate overnight at 41° F. The precipitated enzymes were recovered by centrifugation and dialyzed against a large volume of distilled water in the cold. The recovered enzymatic material was reconstituted to one-half original volume with 0.02 molar phosphate buffer and rendered bacteria free by passage through a Selas filter.

Endocellular enzymes were obtained by treating the cell suspension in a 10 KC Raytheon Sonic Oscillator* for 50 min. The sonic extract was centrifuged twice at 30,000 x g for 20 min to remove cellular debris and then rendered bacteria free by passage through a Selas filter. The bacterial extract was stored at 32° F for periods up to 4 days before use, without serious loss in proteolytic activity.

RESULTS AND DISCUSSION

Frozen chicken pies were stored at 41° F for periods up to 14 days. Figure 1 presents data on the numbers of bacteria per g of chicken pie gravy as a function of time of incubation.

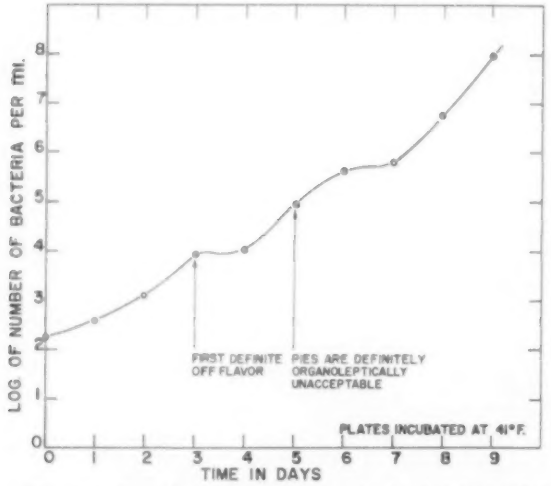
LOG OF NUMBER OF BACTERIA PER ml.
TIME IN DAYS
FIRST DEFINITE OFF FLAVOR
PIES ARE DEFINITELY ORGANOLEPTICALLY UNACCEPTABLE
PLATES INCUBATED AT 41°F.

Figure 1. Bacteria counts on chicken pies incubated at 41° F (5° C).

The first definite off-flavors and aromas began to appear on about the third day of storage at 41° F. The number of bacteria was of the order of 10,000 per g. By the fifth to sixth days the pies were very definitely organoleptically spoiled. The bacterial population ranged from 100,000 to 1,000,000 microbes per g. Thus, at the time spoilage became incipient, no great number of bacteria were present. Even at spoilage, the total number of bacteria per gram of chicken pie was still comparatively low and did not reach the vast numbers ordinarily associated with spoilage and which the extreme organoleptic changes would suggest. In general, 24 hr are required for the frozen pie to thaw completely at 41° F.

The proteolytic activity of water extracts of the gravies of these pies was also determined. Figure 2

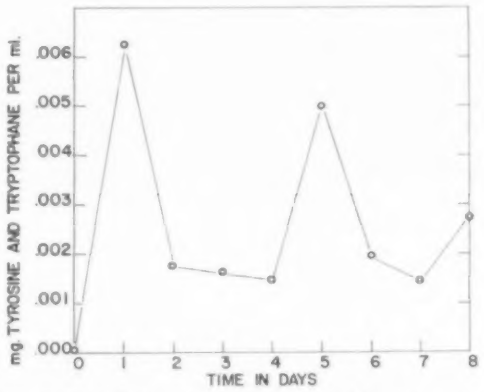

Figure 2. Proteolytic activity of water extracts of the gravies.

shows the typical data; characteristically, the curve had a biphasic character with a peak of maximum proteolytic activity at one to two days. Friedman, Nelsen and Wood *(7)* obtained similar results with *Bacterium linens*. A second peak of proteolytic activity occurred at 5 to 6 days. It was noted that the first peak of proteolytic activity occurred at a time when the bacterial population was only a few thousand bacteria per g. Apparently, potent extracellular proteolytic enzymes were liberated in the growth menstruum to break down the complex proteins so that the bacterial cells could obtain the necessary nutritional requirements. The smaller peptide units probably provided ample supplies of soluble nutrients for the growth of a considerable number of bacteria.

A culture of *Pseudomonas fluorescens* that grew vigorously at low temperatures was isolated from a defrosted chicken pie. The culture was markedly proteolytic, lipolytic and saccharolytic. At 32° F the lag phase of the culture was only 24 hr long, and at 50° F the lag phase was less than 8 hr. The culture did not grow at all at 98.6° F. Olson, Parker and Mueller *(9)* observed that the growth of psychrophiles in terms of numbers increased as the temperature increased at least to 71° F.

Chicken pies were inoculated with aliquots of bacteria-free broth growth medium in which the culture of *Ps. fluorescens* had been grown for 120 hr at 50° F. Figure 3 presents photographs of these pies and a control pie after incubation for 3 days at 41° F following treatment. Four and a half ml of sterile water were added to the control pie. Separation of fat

*Manufactured by Raytheon Manufacturing Company, Waltham, Mass.

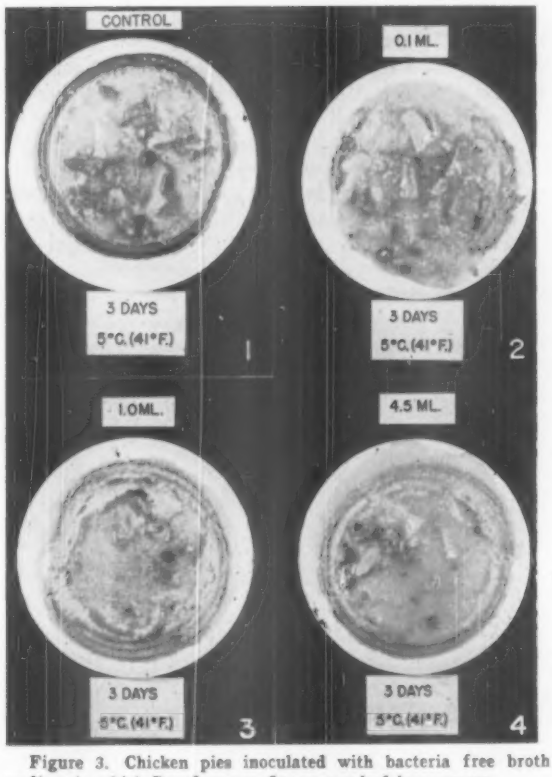

Figure 3. Chicken pies inoculated with bacteria free broth medium in which Pseudomonas fluorescens had been grown.

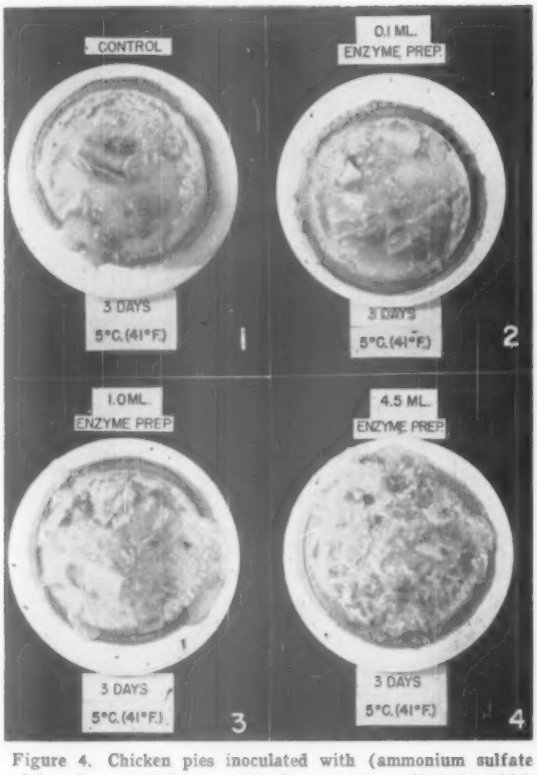

Figure 4. Chicken pies inoculated with (ammonium sulfate precipitated) exocellular enzymes from broth medium in which Ps. fluorescens had been grown.

particles into discrete globules, thinning of the gravies, and curding of the gravies are typical symptoms noted in the broth treated pies.

Aliquots of the "purified" extracellular enzymes were added to chicken pies. The control pie received 4.5 ml of sterile water. These pies were also stored at 41° F for 3 days. Figure 4 shows photographs of these pies after incubation and baking. The damaging effects of these enzymes quite clearly simulate those observed with defrosted merchandise. Normal (control) gravies have a smooth translucent character with no fat separation. As the amount of enzyme preparation added was increased, fat separation and the curdled texture became very noticeable. The characteristic offensive odor of defrost and discoloration of vegetables occurred. The crust also became soggy and discolored.

In view of the apparent high activities of the extracellular proteolytic enzymes from this culture of *Ps. fluorescens*, and the pronounced curding of the gravies accompanying the treatment of chicken pies with extracellular enzymes, it became desirable to determine if the extracellular amylolytic enzymes also had high activities. To determine the amylolytic potency of the extracellular enzymes, aliquots of the cell-free broth medium in which *Ps. fluorescens* had been grown were added to sterile starch gels and the gels then incubated at 32° F and 50° F for 5 days. One per cent of sterile water was added to the control.

The extracellular amylolytic enzymes in the growth medium were indeed very potent. The control starch gel maintained a viscosity of 10,100 centipoises (cp) throughout the experiment. At the dilute concentrations of saccharolytic enzymes available in the broth medium, a viscosity of 7,370 cp resulted with a 1% inoculum after 5 days' storage at 50° F. This amounted to 25% reduction in starch viscosity. At 32° F the viscosity was 8,240 cp with a 1% inoculum after 5 days, or about an 18% reduction in viscosity. Once saccharolytic enzymes were elaborated, low temperatures retarded but did not completely inhibit their liquifying action. The extreme potency of these enzymes is doubtless responsible for one of the most noticeable features of defrost pies, the thinning of the gravy.

Encouraged by these manifestations of extracellular enzymatic activity on the part of this psychrophilic bacterium, attention was directed to the endocellular enzymes.

Figure 5 shows the photographs of chicken pies after inoculation with aliquots of the sonic extract and storage at 34° F for 7 days. The unopened control and opened control pies were used to test the effect of removing the top crust of the pie, stirring the gravy, and replacing the top crust to parallel the treatment given the other pies in this experiment. The other control pie had 1.0 cc of water added to simulate the amount of cellular fluids added to the test pies. One cc of the supernatant cell washing fluid from the

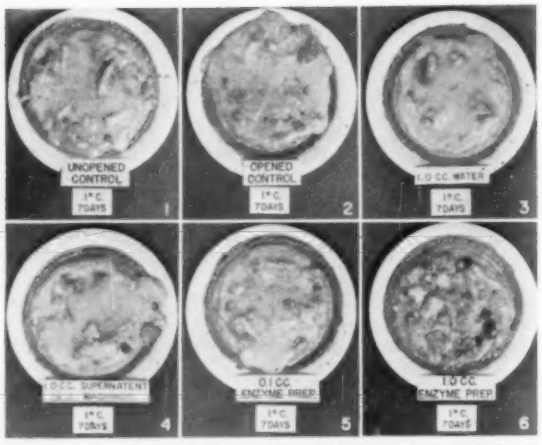

Figure 5. Chicken pies inoculated with cell free sonic lysates of Pseudomonas fluorescens incubated at 34° F (1° C).

centrifugings in the preparation of the cell suspensions was added to a pie as a comparison between the action of endo- and extracellular proteolytic enzymes. The control pies appeared to be satisfactory, but there was a very slight fat separation. During the preparation of these pies it was noted that the fat was not completely emulsified, and therefore that the gravy was not as good as usual. The pie treated with 0.10 cc of bacteria-free bacterial extract showed some fat separation and slight curdling. The pie treated with 1.0 cc of bacterial extract had extreme fat separation and extreme curding of the gravy. A clear, watery or serous fluid also collected in pockets in the pie. The vegetables were markedly discolored. The crusts were soggy, patchy and discolored. There was a distinct "pinking" of the dark meat in the bacterial extract-treated pies. The enzyme treated pies had a marked defrost odor.

Turkey gravy was inoculated with 0.10, 0.5, and 1.0 cc of bacteria-free bacterial extract and then stored at 34° F for 5 days. Figure 6 shows these results. The same liquefaction of the starch noted previously also occurred here. These spots represent the same quantity of gravy applied to the plate and the size of the spot is a reflection of viscosity. The viscosity of the gravy to which 0.1 cc of enzyme preparation had been added was 4,280 cp. The viscosities of

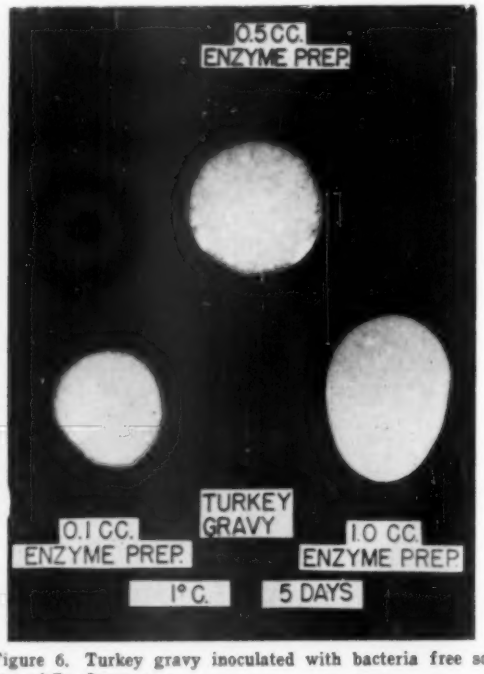

Figure 6. Turkey gravy inoculated with bacteria free sonic lysates of Ps. fluorescens.

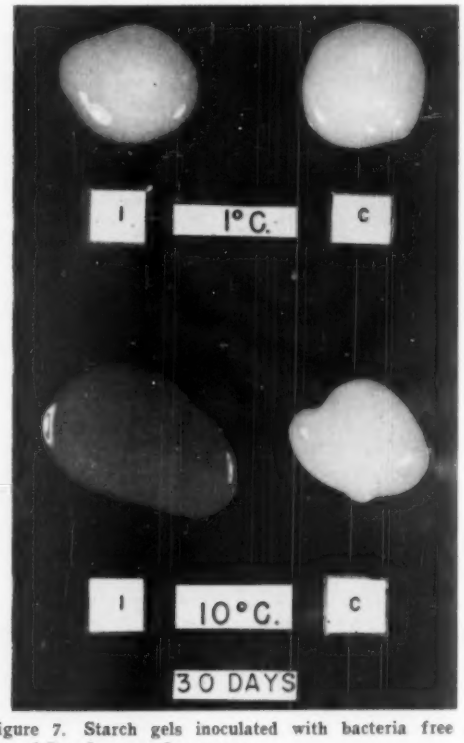

Figure 7. Starch gels inoculated with bacteria free sonic lysates of Pseudomonas fluorescens.

the gravies to which 0.5 cc and 1.0 cc of bacterial extract were added were 1,760 and 890 cp respectively. Although the gravy sample to which 0.5 cc of enzyme preparation was added shows greater curd formation than the sample receiving the larger inoculum, curd formation is apparently an intermediate stage in the destruction of the starch gel. Since the curds are discrete and only a few appear in the sample treated with 1.0 cc of enzyme preparation, destruction of the starch gel is apparently spotty and nodules of starch gel disappear more slowly than general deterioration takes place. Note the definite ring of clear fluid at the edges of the samples inoculated with 0.1 and 0.5 cc of enzyme preparation.

Figure 7 is a photograph of sterile starch gels inoculated with 0.25% of cell-free bacterial extract

and stored at 34° F and 50° F for 30 days. One is the enzyme inoculated test gel and C is the uninoculated control. After incubation at 34° F the viscosity of the control was 8,900 cp; that of the enzyme-treated starch gel was 4,900 cp. After incubation at 50° F, viscosity of the control was 10,500 cp, whereas that of the enzyme-treated starch gel was 31 cp. It was apparent that virtually complete liquefaction of the starch gel resulted at 50° F and substantial liquefaction at 34° F.

The endocellular enzymes have been investigated at 10° F using these same techniques, and similar effects of a smaller magnitude were observed. Thus, once the enzymes of psychrophilic bacteria are produced and liberated in the product, lowering the temperature will decrease the rate at which damage occurs to the product, but will not stop it. In conclusion, the words of the late Dr. Carl R. Fellers *(6)* are appropriate. "In any case, the consuming public should have assurance that the freezing, storage and transportation are effective and under control and that frozen foods offered for sale have not been previously thawed."

SUMMARY

The development of psychrophilic bacteria in frozen foods stored at defrost temperatures has been observed. The liberation of potent extracellular proteolytic enzymes in the growth menstruum at times and temperatures which might have been expected to afford the product some protection has been demonstrated. The development of off-flavors and odors in defrosted chicken pies in advance of the development of great numbers of psychrophilic bacteria has been noted. A psychrophilic culture of *Pseudomonas fluorescens* with marked enzymatic activities was isolated. The proteolytic and saccharolytic activities of both extra- and endocellular enzymes from this microbe was demonstrated. Previously observed defrost effects were duplicated by the addition of these enzymes to normal chicken pies. It was demonstrated that once the enzymes of psychrophilic bacteria are elaborated and liberated in the growth menstruum, lowering the temperature will slow their action, but will not stop it, and definitely will not remedy damage already done. Therefore, frozen foods can be said to have a built-in indicator of defrost. This indicator is the effect of psychrophilic bacteria and their enzymes on the physical characteristics of the product.

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Functional Properties and Flavor of Eggs Laid by Hens on Diets Containing Different Fats[a]

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NUMEROUS REPORTS have been published on the effect of different fats in the diet of laying hens upon the nature of the fat in the egg yolks. Some of these have been cited in a recent article by Ostrander, Jordan, Stadelman, Rogler and Vail *(15)*. There is a dearth of information available, however, upon the effect, if any, of different fats in the hens' feed upon the functional properties, such as foaming characteristics, emulsifying ability, or thickening property, of the eggs produced.

King, Whiteman, and Rose *(12)*, who studied certain physical and chemical properties of eggs from five groups of hens on different rations, concluded that the chemical and physical measurements of the eggs and the volume and tensile strength of sponge cakes baked from them indicated that the different diets did not affect the properties of the eggs. The diets differed chiefly in the substitution of a certain percentage of soybean oil cake meal, peanut oil cake meal, linseed oil cake meal, or combination of fish meal, meat meal and dried buttermilk for part of the basal feed mixture.

Reports upon the flavor of eggs in relation to fat in the diet have dealt chiefly with feeds containing fat of pronounced flavor such as fish meal *(1)*, cod liver oil *(1)*, or diets containing linseed oil *(3, 8, 23)*.

In the study herein reported, functional properties of eggs from hens on rations containing different fats were compared with respect to stability of whole-egg foams and to leavening power in whole-egg sponge cakes. In addition, some of the eggs were used in a taste panel study to observe possible differences in flavor.

EXPERIMENTAL

Eggs. The eggs used were from the 3 groups of Leghorn hens, 15 hens per group, used by Ostrander and associates *(15)* to study the influence of fat in the diets upon egg yolk fat. For the first 8 days, all hens were fed the same stock ration given the flock from which the 45 hens were taken. After this base period, hens in one group were fed the stock ration supplemented with 10% corn oil; a second group received the stock ration supplemented by 10% beef tallow; the third group was continued on the regular stock diet.

Eggs were gathered daily and each was identified with hen number and date of lay. As soon as the eggs were brought from the Poultry Science Department to the Foods and Nutrition laboratory, they were placed under refrigeration. Eggs from the 3 groups of hens were handled alike throughout the investigation.

The first 3 to 5 eggs collected from each hen during each of 4 periods of the experiment—base week and 2, 4, and 8 weeks after the corn oil and beef tallow were incorporated into the feed—were reserved for chemical analyses of the egg yolks *(15)*; remaining eggs were available for functional or flavor tests.

During the first 4-week period after the 3 groups of hens were put on the different diets, eggs from each group, not used immediately for chemical or functional tests, were broken out and separated. Whites and yolks were blended separately, strained, then recombined, weighed out into small packages, labelled, frozen, and held in a freezer maintained at a temperature of about −5° F to 0° F (−20.5° C to −18.0° C) for later use. During the last few weeks of the study, shell eggs not needed for immediate tests were washed, air-dried, put in cartons and sprayed.[b] These were held for 3 months or longer in a refrigerator of about 27 cu ft capacity at a temperature around 35° F (1.7° C) in an atmosphere of high relative humidity.

Unless otherwise stated, data obtained on each egg used in the tests on functional properties included weight of shell egg, albumen height, weight of white and weight of yolk. Since white and yolk were separated by use of a small household-type separator, the weights recorded represented the respective amounts of yolks and whites as obtained in practical use. The white-yolk ratio of each egg was calculated by dividing weight of white by that of the yolk.

Stability of whole-egg foams. Purpose of this phase of the study was to compare the stability of whole-egg foams made from eggs laid by the 3 groups of hens and to determine whether from such a test the performance of eggs in sponge cakes might be reliably predicted.

Except in the first 3 of the 6 series, 48 g of egg magma were put into a weighed, pint-size plastic cup with slightly rounded bottom, brought to a temperature of 25° C, then whipped for 5 min at Speed 10 (highest speed) by a Hamilton-Beach household-size electric mixer. Weight of the foam in the cup after the beater was removed was recorded. The cup of foam was covered with a watch-glass and allowed to stand at room temperature for 60 min. At the end of that period, the liquid which had separated was carefully poured into a weighed graduated cylinder and its weight and volume recorded. The percentage of drainage was calculated from weight of the foam and weight of the drained liquid. Six series of tests were made.

Series 1. Individual eggs were used as they came from the shell. These eggs were laid during the base week and 3 days of the first week after the feeding of the corn oil and beef tallow supplemented diets was begun. Weights of the broken-out eggs varied from 45.5 to 62.9 g. Data on the albumen height and the white-yolk ratio for each egg were examined for any relationship of these factors to percentage of drainage.

Series 2. Individual eggs laid during the same period as those in Series 1 were used. Since, throughout the study, the effect of yolk differences upon functional properties of the eggs was of concern, whites of all eggs used in each replication of this series were blended. Each yolk was weighed separately into a cup and to it was added a portion of the blended whites so that the ratio of white to yolk was 30 to 18 (or 1.66).[c]

Series 3. Eggs laid after the hens had been on the special diets for 3 weeks were treated as in Series 2.

Series 4. These eggs were laid during the 4th week after the hens had been fed the special rations. In each replication,

[a] Journal Paper 1571 of the Purdue Agricultural Experiment Station, Lafayette, Indiana.

[b] The egg washing solution was a "Chore-Time Formula CT" solution; the spray was "Sta-Good Egg Spray."

[c] The "average" egg used in experimental cookery is considered to consist of 30 grams of white and 18 g of yolk *(14)*; hence, this ratio was used.

whites of the eggs from a given group were blended and strained; yolks were similarly treated. Samples for whipping consisted of 30 g of blended whites and 18 g of the corresponding blended yolks.

Series 5. Eggs for this series were laid during the 6th and 7th weeks that the hens were on the fat-supplemented diets. When broken out, each egg was scored by comparing it with a U.S.D.A. chart *(22)* instead of measuring its albumen height. The eggs were then prepared as described for Series 4. Samples from these same eggs were used for making sponge cakes in Cake Series 1.

Series 6. Eggs for this series were laid between the 7th week of the special diets and the close of the experiment. They were held under refrigeration as described under the section on eggs. Since the whites and yolks of these stored eggs were not readily separated, the unseparated eggs from each group were blended and 48-g portions used for the stability of foam tests.

Sponge cakes. Small sponge cakes, one-egg size (that is, 48 g whole egg magma per cake), were made according to proportions and methods found satisfactory in previous studies in this laboratory *(10)*. The mixing was done in a constant-temperature room on a Kitchen Aid Model 3C electric mixer. Eighty grams of batter were weighed into a baking pan with slightly sloping sides, size at the top 6¼ by 3⅜ in and 2¼ in deep. The bottom of the pan was lined with a piece of lightweight aluminum foil. The batter was baked for 18 min in a reel-type electric oven regulated for 350° F (176° C). Upon removal from the oven, pans were inverted on a rack, cooled, and the cake volumes measured by seed displacement in a volumeter (National Manufacturing Company). Cakes were then cut and observations made on the general appearance, color, grain, and cell wall thickness.

There were 3 series of cakes, made as follows:

Cake Series 1. For this series, 25 cakes were made from freshly-laid eggs from each of the 3 groups of hens. The same number of cakes from each group of eggs was made on any one day of baking, but the order of mixing was randomized. Eggs from all the hens laying during the period in which these cakes were baked were represented in the 25 replications. Cakes in this series were made from portions of the same eggs used for Series 5 of the tests on stability of foams. (Methods of preparation of the egg samples are described under Series 5, Stability of Foams).

Cake Series 2. Cakes in this series were made from shell eggs laid after the 6th week of the feeding of the special diets; the eggs had been held under refrigeration from 6 to 8 months. Since, in attempting to separate white and yolks of these eggs, yolk membranes frequently broke, unseparated whole eggs for any one group were blended, then weighed into 48-g portions. Otherwise the procedure was the same as for Cake Series 1.

Cake Series 3. These cakes were made from frozen eggs which had been stored in the freezer for 8 months or longer. The eggs had been laid during the first 3 weeks of the experiment. Samples used were from composites of yolks and whites in the proportion of 30 g white to 18 g yolk.

Flavor tests. Eggs used for flavor tests were taken from those laid after the hens had been on the diets 8 weeks or longer. A composite sample of broken-out eggs from each group of hens was scrambled. Samples were cooked in glass beakers in a water-bath, stirred with glass rods during cooking, and cooled to room temperature for tasting. Samples were presented in pairs to the six-member taste panel. The sample of eggs from hens on the regular stock ration was considered "standard"; samples from groups fed corn oil and beef tallow were compared with the standard. All tasting was done in the taste-panel laboratory; each booth was equipped with color filters to eliminate differences in color of egg samples.

Two series of flavor tests were carried out. Freshly laid eggs were used for one series. For the second, eggs laid by the same hens but which had been held in refrigerator storage for 3 months were used.

Observations on color. Any differences in color of the egg yolks, the foams, the cake batters and baked cakes, and of the scrambled eggs used in the flavor tests were noted, but no objective color measurements were made.

Statistical analyses of data. Analyses of variance and F-tests *(21)* were made on the data on the percentage of drainage in the stability of foam experiments and on the volumes of the sponge cakes. For testing the significance of differences among averages, multiple range tests were used *(5, 13)*. Coefficients of correlation calculated were those between percentage of drainage of foams and (a) albumen height of the eggs and (b) white-yolk ratio of eggs in Series 1 of the foam tests, and the correlation between cakes volumes of Cake Series 7 and the drainage of foams. The Chi-square test was applied to the flavor test data.

RESULTS AND DISCUSSION

Stability of foams. Percentages of drainage from egg foams in the various series are shown in Table 1. In neither Series 1 nor 2 were differences among the 3 groups statistically significant. Since eggs in these series were laid during the base week and the first few days after the hens were put on the special diets, little difference might be expected among the groups that could be attributed to the diets. For Series 1, wherein individual eggs were used as they came from the shell with no adjustment made as to the proportion

TABLE 1

Average percentage of drainage from whole-egg foams of eggs from three groups of hens on different diets: stock ration plus 10% corn oil (A); stock ration plus 10% beef tallow (B); stock ration only (C)

Series No. and treatment of eggs	Average percentage of drainage			Significance of difference[1] between averages of		
	A	B	C	A and B	A and C	B and C
	%	%	%			
Series 1 Individual eggs[2]	58.0	60.8	62.5	n.s.	n.s.	n.s.
Series 2 Individual yolks to blended whites[3]	60.0	63.9	61.1	n.s.	n.s.	n.s.
Series 3 Individual yolks to blended whites (white-yolk ratio 30:18)[4]	54.3	60.9	58.8	....*	n.s.	n.s.
Series 4 Individual yolks to strained blended whites (white-yolk ratio 30:18)[4]	49.8	57.3	54.2	....*	n.s.	n.s.
Series 5 Composite of yolks to composite of whites (white-yolk ratio 30:18)[5]	43.7	58.3	55.5	....**	....**	n.s.
Series 6 Composite of unseparated eggs[6]	47.6	54.4	52.3	....**	....**	n.s.

[1] Significance of differences: n.s. not significant, * significant at 5% level, ** significant at 1% level.
[2] Eggs laid during base week of experiment; 14 samples in each average.
[3] Eggs laid during base week; 18 samples of A, 12 of B, and 13 of C in average.
[4] Eggs laid during third week after hens were put on the special diets; 14 samples in each average for Series 2; 16 samples, Series 3.
[5] Eggs laid during sixth and seventh weeks of special diets; 22 composite samples in each average; samples from these same eggs used in Cake Series 1.
[6] Eggs laid between seventh week and end of the experiment and refrigerated 6 to 8 months; 26 samples of A, 35 samples of B, 47 samples of C, in respective averages.

TABLE 2

Average volumes of sponge cakes made with eggs from three groups of hens on diets differing in fat content: stock ration plus 10% corn oil (A); stock ration plus 10% beef tallow (B); stock ration only (C)

Series No. and treatment of eggs	Average cake volume[1]			Significance of difference[2] between averages of		
	A	B	C	A and B	A and C	B and C
	ml	*ml*	*ml*			
Series 1						
Freshly laid eggs[3]	467	439	469	....**	n.s.	....**
Series 2						
Eggs held under refrigeration 6 months or longer[4]	536	497	528	....**	n.s.	....**
Series 3						
Frozen eggs[5]	520	504	513	....**	....*	....*

[1] Volume expressed as ml cake per 80 g batter.

[2] Significance of differences: n.s. not significant, * significant at 5% level, ** significant at 1% level.

[3] Eggs laid during 6th and 7th weeks after hens were put on the special diets; composite of yolks combined with composite of corresponding whites with white-yolk ratio 30:18. There were 25 cakes represented in each average.

[4] Eggs laid after hens had been on special diets 8 weeks; number of cakes represented in averages: 17 for A, 35 for B, 56 for C. The number of bakings from A group was limited because during the later weeks of the experiment many of the hens quit laying.

[5] Eggs used in Series 3 were laid during the first 3 weeks after hens were put on the special diets; eggs had been in frozen storage for about 8 months. Number of cakes represented in the averages: 58 for A, 47 for B, 41 for C.

of white to yolk, no significant correlation was found between percentage of drainage and white-yolk ratio or between percentage of drainage and albumen height of the eggs. (The white-yolk ratios for the individual eggs ranged from 1.54 to 2.12, and the albumen heights from 4.6 to 9.0.)

In Series 3 and Series 4, the average percentage of drainage of foams from the group of eggs from hens on the beef tallow diet was significantly greater than that of the corn oil group. In Series 5 and Series 6 there was also a highly significant difference between the averages of the corn oil and the stock ration groups as well as between the corn oil and beef tallow groups. Although in every series except Series 1, the average percentage of drainage of foams of the beef tallow group was higher than that of the stock ration groups, the difference did not reach a statistically significant level. Eggs used in Series 5 and Series 6 were laid after the hens had been on the experimental diets 6 weeks or longer. Since the chemical analyses *(15)* of yolks of the eggs laid during this period indicated a significant difference among the iodine values of the ether extracts of the yolks of the 3 groups of eggs, there might be some relation between egg yolk fat and percentage of drainage from foams.

As the experiment progressed, egg yolks of the corn oil group, and, consequently the foams, became much lighter yellow than corresponding eggs and foams from the beef tallow and stock ration groups.

Sponge cakes. Averages of the volumes of cakes of Series 1, made from eggs laid by the 3 groups of hens, are shown in Table 2. The difference between the volume averages of cakes from the corn oil and the stock ration groups was not statistically significant, but the average volume of cakes of the beef tallow group differed from that of both the other groups of cakes at the 1% level of significance. Cakes made from eggs from the beef tallow fed hens were consistently smaller in volume.

Although each cake was made from 30 g of an egg white composite and 18 g of the corresponding yolk composite, the possible influence of the scores of the eggs and of the original white-yolk ratios of the eggs in each group was considered. The means of the scores and of the white-yolk ratios for each of the 3 groups of eggs were compared. Neither means of the scores nor of white-yolk ratios were significantly different. Hence, any characteristics of the eggs in the 3 groups associated with their scores or with their original white-yolk ratios do not offer a sufficient explanation for the differences observed in the cake volumes.

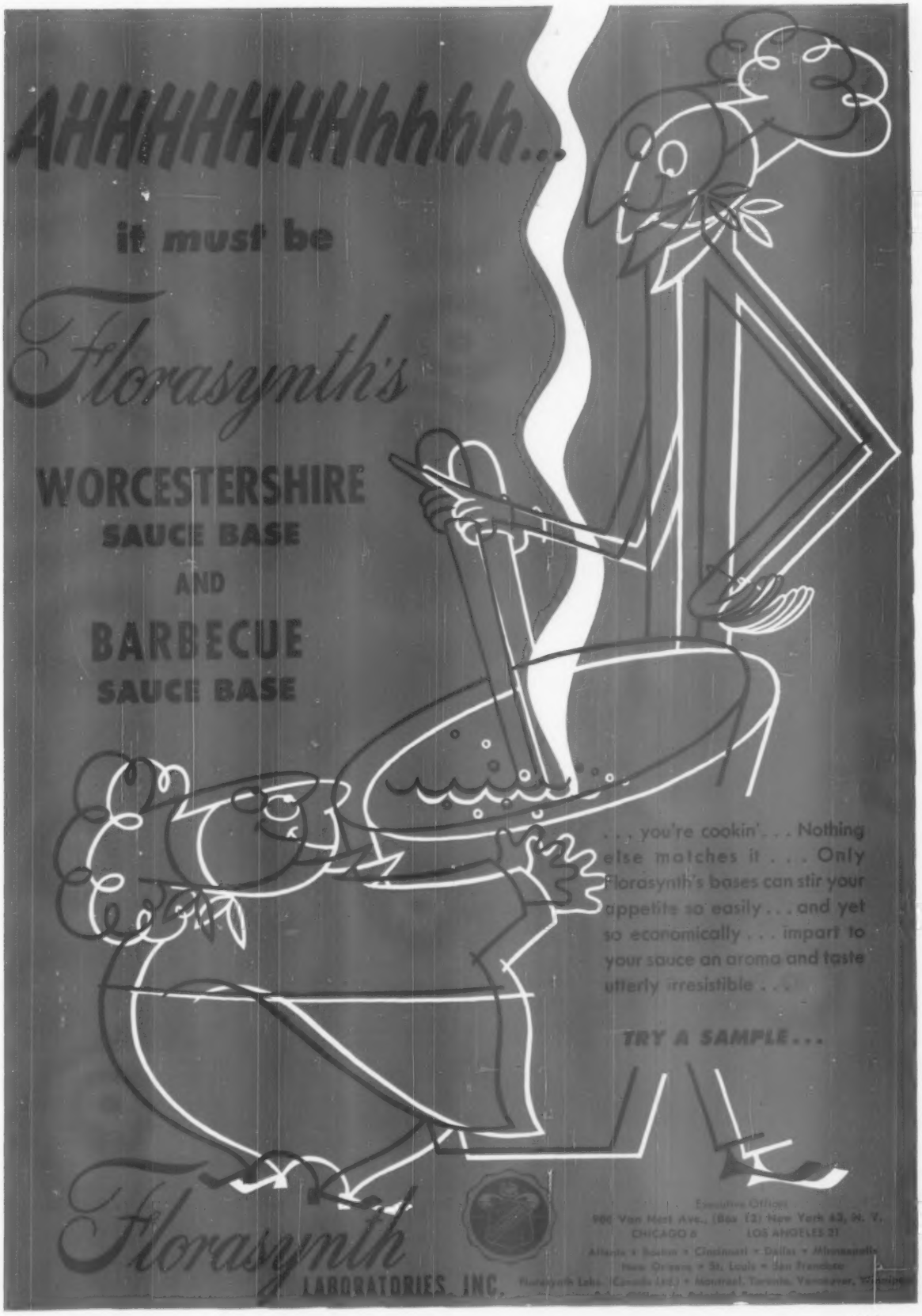
The coefficient of correlation between cake volume and percentage of drainage from foams of samples of the same eggs that were used in the cakes was −0.67. Although this r-value was statistically highly significant, it is not high enough to predict cake volume that may be expected in individual cases.

Average volumes of cakes baked from eggs held under refrigeration for 6 months or longer (Cake Series 2) are also presented in Table 2. Although for each group these averages were somewhat greater than corresponding means for Series 1, the relationship among the 3 groups was essentially the same.

Cakes in Series 3 were baked from frozen eggs. In this series the difference in average volumes of cakes from the corn oil and beef tallow groups was significant at the 1% level; other differences were significant at the 5% level. It should be noted that the eggs used in these cakes were laid when the hens had been on the special diets only a short time.

All of the cakes were of good quality even though they differed in some respects. There was a marked difference in the crumb color of cakes from the 3 groups of eggs; cakes from the corn oil group were much lighter yellow. This difference became more marked in cakes made from eggs laid after the feeding of the experimental diets had been underway more than 6 weeks. The crumb of cakes made from eggs of the beef tallow group laid in the later weeks of the experiment was somewhat lighter in color than that of the stock ration group for the same period. Crusts of cakes of the corn oil group differed in both texture and color from those of cakes from the two other groups.

In all 3 sets of cakes, the average volume of cakes of the beef tallow group was significantly smaller than that of either of the other groups; the reason is not readily apparent. In each series of bakings, and in each series of foam tests, eggs from each of the 3 groups were comparable in age and had received the same treatment. The higher percentage of drainage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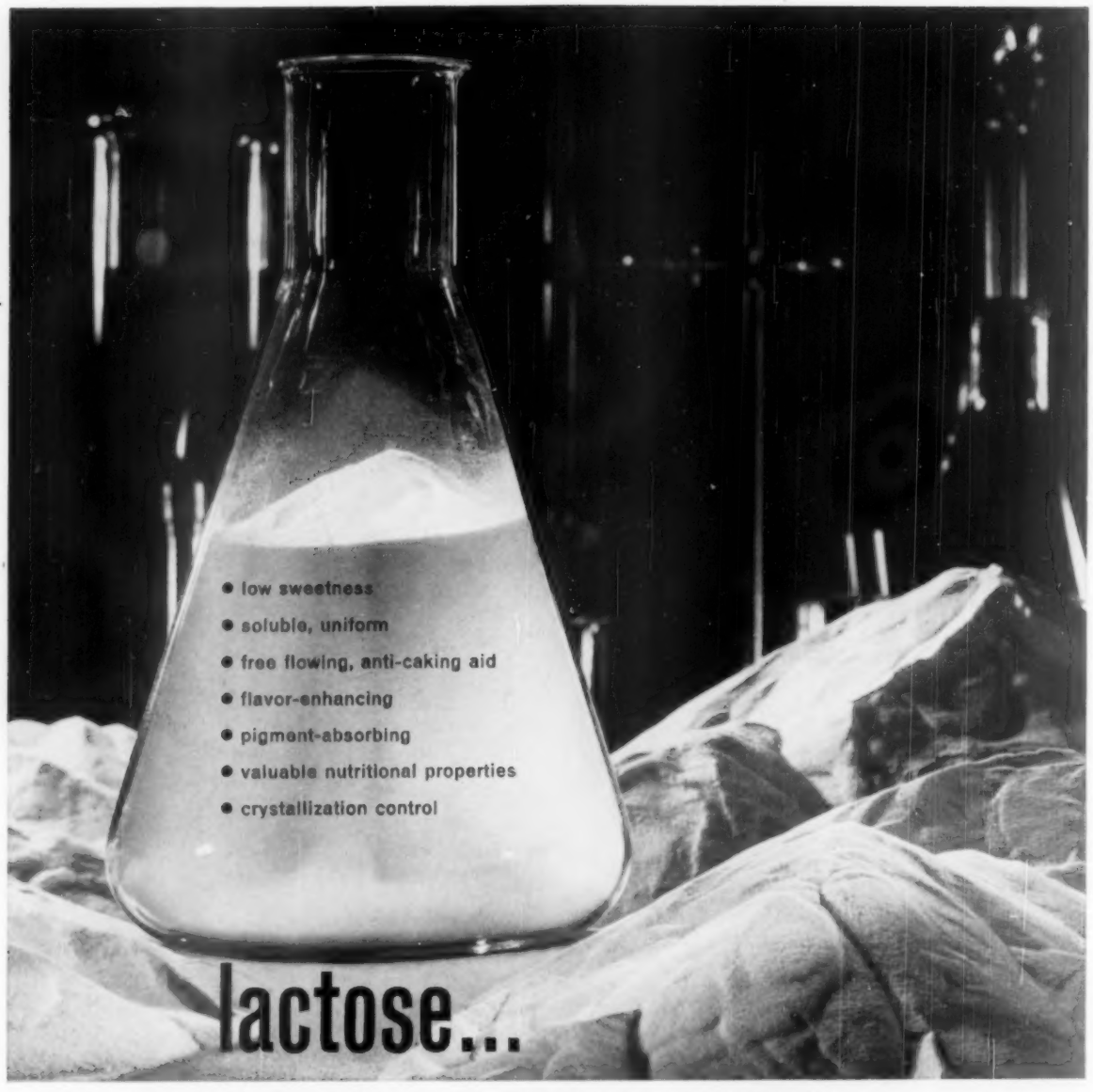

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from foams of the beef tallow group indicated a tendency to less stability even though they did not differ to a statistically significant degree from foams of eggs from hens on the stock ration. Foams of the corn oil group had the lowest average percentage of drainage, but cake volumes were not always the highest. It might seem that there is an optimum degree of foam stability conducive to larger cake volume. Pyke and Johnson *(17)*, who compared rate of drainage of whole-egg-sugar meringues with sponge cake volumes, implied that a moderately stable meringue produced a cake of better volume than a more slowly draining meringue. However, these workers did not state how a somewhat more rapidly draining foam would affect the stability of the cake batter and the ultimate volume of the baked cake.

Whether or not there is some relationship of foam drainage and sponge cake volume to the degree of saturation of the fatty acids in egg yolk can only be surmised. The average iodine value for the ether extracts of the eggs from the hens on the stock ration after 8 weeks was 76; for the group fed the corn oil supplemented diet it was 82; and for the group fed the beef tallow supplemented diet it was 72 *(15)*. The appreciable difference between iodine values of the ether extracts of egg yolks from the corn oil and beef tallow groups suggests a possible relation between the nature of the lipids of the eggs and their functional properties. On the other hand, despite a considerable difference between iodine values of the ether extract of eggs from the corn oil and stock ration groups, the volumes of cakes made from the eggs of these 2 groups differed but slightly. The difference between the percentage of drainage of the foams of these 2 groups was not as great as that between the corn oil and beef tallow groups. Perhaps differences in only certain lipids of the egg are involved in differences in functional properties.

Farrand *(6)* has suggested that the stability of egg-sugar foams, such as are used in making sponge cakes, is related to the size distribution of the bubbles. The possible influence of the nature of the lipids of the egg yolk upon the number and size of air cells and upon foam stability when whole egg is whipped under given conditions merits detailed study.

As far as the authors are aware, in no reported studies on sponge cakes made with fresh eggs has consideration been given to the possible relation of the composition or condition of egg yolk fat to the stability of the egg foam; however, Joslin and Proctor *(11)* have attributed lack of foam volume in whipping tests with reconstituted whole egg powders to breakdown during dehydration of the natural fat emulsions of fresh whole eggs (with the consequent formation of a surface of free oil), and the failure of the original emulsion to re-form upon rehydration of the powder.

Since it is well established that the fatty acid composition of the phospholipids of egg yolk *(18, 19, 23)*, as well as the lipids as a whole, can be affected by the diet of the hen, it follows that all egg yolks are not identical with respect to the nature of their fat. The presence of considerable amounts of phospholipids and lipoproteins in egg yolk *(7, 9)* and the effectiveness of certain of these for such functions as emulsifiers *(20)* suggest a relationship between the nature of the egg yolk lipids and various functional properties of the egg. Such a possibility would seem to merit future investigation.

Flavor tests. Results of the flavor tests are shown in Table 3. In the tests for the freshly laid eggs the taste panel could distinguish between the corn oil and stock ration groups; when the eggs of the beef tallow group were compared with the stock ration group, the panel had a higher percentage of "wrong" answers than "right", having mistaken the eggs of the beef tallow group for the "standard" samples. This might indicate that there was a distinguishable difference but that some quality other than flavor influenced the judging.

TABLE 3

Results of flavor tests by taste-panel, using a two-sample procedure

	Eggs of corn oil group compared with stock ration group		Eggs of beef tallow group compared with stock ration group	
	Fresh eggs	Stored eggs	Fresh eggs	Stored eggs
Total number of tastings	130	139	221	169
Number of "correct" selections	77	75	65	110
Percentage "correct" answers	59.2	53.9	29.4	65.0
Adjusted Chi-square value	4.07*	0.36	.....	14.79**
Number of "correct" selections in excess of expectancy in standard deviation units[1]	2.10*	0.92	.....	3.92**

[1] See reference, Byer and Abrams *(2)*.
* Significant at 5% level.
** Significant at 1% level.

After the eggs had been stored in a refrigerator for 3 months, the panel found no significant difference between the corn oil and stock ration groups, but a highly significant difference between the beef tallow and stock ration groups. Evidently there was no marked off-flavor, such as rancid flavor. Comments of the judges were repeatedly to the effect that they really could tell no difference, but assumed that one was the "standard".

Samples of the corn oil group were so much lighter yellow in color than those from either of the other groups that it was necessary to use a colored light in order to mask the color of the samples presented to the taste panel.

Color of products. As was pointed out, the cakes, foams, as well as scrambled eggs made with eggs from the corn oil group were much lighter in color than similar products made from eggs of beef tallow and stock ration groups. Just what factors contributed to this difference in intensity of color in the egg yolk pigments are not known. It has been established that xanthophylls in the diet are important in the pigmentation of body fat and skin of chickens and in the egg yolk of laying hens *(16)*. In a recent report on factors that probably influence pigmentation in broilers (and presumably, also, in egg yolk) Williams, Davies, and Couch *(24)* stated that added fat in the diet led to the consumption of somewhat less total feed, hence of less xanthophylls. They also reported that diets containing a high percentage of corn oil, soybean oil, or cottonseed oil, depressed pigmentation to a greater

extent than diets having an equivalent amount of animal fat. Another factor suggested by these workers that possibly affected deposition of xanthophylls was the degree of unsaturation of a particular fat. Their data also indicated that increase in the percentage of free fatty acids in a given fat in the diet led to reduction of pigmentation.

Perhaps some of these factors suggested by Williams and associates *(24)* were responsible for the less intense pigmentation of the yolks of the hens receiving the corn oil supplemented diet.

SUMMARY

Eggs from three groups of Leghorn hens on diets containing different kinds of fat were compared with respect to stability of whole-egg foams as measured by the percentage of drainage in a one-hour period, with regard to leavening power as indicated by the volumes of whole-egg sponge cakes, and with respect to flavor. The diets for the three groups of hens were, respectively, a stock ration supplemented with 10% corn oil, the same stock ration supplemented with 10% beef tallow, and the stock ration only.

Under the conditions of this study the results obtained were as follows:

During the period that the three groups of hens were fed the same diet, no significant differences were found among the foams from the eggs of the three groups. After the hens were on the experimental diets for 3 weeks or longer, the percentage of drainage from the foams of eggs of the beef tallow group was significantly greater than that from the eggs of the corn oil group; and, after 6 weeks, foams from both the beef tallow and stock ration groups had higher percentages of drainage than those from the corn oil group.

Whole-egg sponge cakes made from the eggs of the beef tallow group were significantly smaller than similar cakes made from the other two groups of eggs.

Flavor tests on freshly laid eggs showed distinguishable differences among the three groups; however, after 3 months storage under refrigeration, no significant difference was found between eggs of the corn oil and stock ration groups, but there was a highly significant difference in flavor between the beef tallow and the stock ration groups.

Products made from eggs of the corn oil group were noticeably lighter in color than similar products from the other two groups of eggs.

Inasmuch as the iodine values of the ether extracts of the egg yolks from the three groups of hens were found to differ significantly, the possibility of a relationship between the nature of the fat of the egg yolk and the functional properties of the egg was suggested.

Acknowledgments

Grateful acknowledgment is made to Mrs. Julia McGlasson Frohreich and Mrs. Ruby Wilson for laboratory assistance in the making of products for the functional tests; to all the members of the taste-panel for their services; and to Professor S. R. Miles for advice on the statistical analysis of the data.

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The Determination of Soluble Solids in Citrus Juices. I.

The Effect of Non-sucrose Components on Refractometer Values[a]

(Manuscript received May 15, 1960)

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THE GENERAL PURPOSE of this study is to develop improvements in the accuracy of refractometric and densimetric methods for the determination of true soluble solids in citrus juice products. Current methods of use of the refractometer and the Brix hydrometer with single strength juices produce results in fair agreement with each other, but neither has been satisfactorily correlated with true soluble solids contents of the juices. The graduated scales of both instruments were developed from pure sucrose solutions in water, and have been adopted by the citrus industry without allowing for the effects of non-sucrose components of the juices, other than the effect of citric acid upon refractive index. Maturity standards and quality grades for single strength juices have been set up with "solids" contents based on Brix hydrometer values, giving legal status to the method.

Since concentrated juices are too viscous for the determination of density with hydrometers, the refractometer has become practically the sole instrument for determining the "solids" concentration in such products. Grades and standards have been adopted by various regulatory agencies, with "solids" content based on refractometer sugar scale values corrected for citric acid, the only non-sucrose component for which adequate information was available. The refractometer has been useful for the purpose of adjusting concentration and for judging compliance of products with such standards, but not when used for determining their true soluble solids contents. For the instrument to serve successfully for the latter purpose, allowance must be made for non-sucrose components other than citric acid.

The first part of these investigations, reported here, is concerned with the study of the magnitude of the effects on refractometer sugar values of each of the major components of citrus juices and the development of corrections for them. In a second paper, corrections will be applied to refractometer values of a series of citrus juice concentrates whose compositions have been determined by analyses, and corrected values will be compared to soluble solids as determined by vacuum drying.

LITERATURE ON THE REFRACTOMETRY OF MATERIALS FOUND IN CITRUS

Sucrose. The refractometer has served many years as the classical instrument for the estimation of soluble solids in solutions containing sucrose as the principal solute. Accuracy and precision of the refractometric method for determining soluble solids have been the subject of intensive study. Browne and Zerban *(2)* indicate that differences in refractive indices at 20° C of 10% sucrose solutions, as reported by six reputable investigators, amount to as much as 4 units in the 4th decimal place, which is roughly equivalent to 0.2% sugar.

Charles and Meads *(1)*, using pure sucrose solutions with utmost caution to eliminate sample variables, compared the precision and accuracy of two refractometers, the Bausch and Lomb Precision[d] and the Zeiss.[d] The data indicated that readings of pure sugar solutions can be repeated with the Precision refractometer within an error of about 0.03% solids, while with the Zeiss refractometer the error is about twice as great. They reported that each instrument was accurate to within 0.5% solids at sugar concentrations up to 70% solids.

Reducing sugars. Analyses reported by Chatfield and McLaughlin *(5)*, Roberts and Gaddum *(16)*, Widdowson and McCance *(22)*, Curl and Veldhuis *(6)*, and McCready, Walter, and Maclay *(13)* indicate the presence in orange juice of no appreciable quantities of sugars other than sucrose, glucose, and fructose. Curl and Veldhuis found that the ratio of sucrose to reducing sugars did not change significantly during storage for a year at 40° F.

In 1931 Macara *(11)* published corrections for invert sugar, glucose, and citric acid which were designed to convert refractometer values of jams to true soluble solids. Tables of the refractive indices of fructose solutions were published in 1932 by Jackson and Mathews *(2, 9)*. A similar table published in the 1955 *Official Methods of Analysis (1)*, without credit for its source, presents values differing from the above by as much as .00003 units, equivalent to about 0.03%

[a] Presented at the Twentieth Annual Meeting of the Institute of Food Technologists, May 17, 1960, San Francisco, California.

[b] Now at Redstone Arsenal.

[c] One of the laboratories of the Southern Utilization Research and Development Division, Agricultural Research Service, U. S. Department of Agriculture.

[d] The mention of trade products does not imply that they are recommended by the Department of Agriculture over similar products not mentioned.

sugar. Tables of the refractive indices of glucose and invert sugar were published in 1944 by Zerban and Martin *(23)*, and reproduced in the 1955 *Official Methods of Analysis (1)*. The refractive indices of glucose solutions were slightly lower than those of sucrose solutions of equivalent concentration, while those of fructose solutions were still lower. The refractive indices of invert sugar solutions (equimolar glucose and fructose) were lower than the average for glucose and fructose. Corrections must be added to refractometer sugar scale values for these sugars to indicate true soluble solids.

Citric acid. Citric acid has an index of refraction considerably different from that of sucrose. Corrections published by Macara *(11)* in 1931 have already been mentioned. In 1932 McRoberts *(14)* published corrections for citric and tartaric acids in fruit juices. In 1939 Stevens and Baier *(17)* published comprehensive data on density and refractive index of citric acid in aqueous solution. They made up a series of solutions of citric acid of known concentrations and experimentally determined their densities and refractive indices. They found the hydrometer Brix of the solutions to be higher, and the solids by refractometer to be lower than true dissolved solids. By plotting results they developed curves from which were derived tables of corrections to be added to refractometer sugar scale values to give both true dissolved solids and equivalent hydrometer Brix values. The table for correcting refractometer readings to degrees Brix is currently used throughout the citrus industry.

Mineral and amino compounds. The effect of mineral and amino components on the refractometric estimation of dissolved solids was investigated by Stanek in 1909 *(18)*. He presented refractometer sugar scale values for 5, 10, and 25% water solutions of betaine, and sodium and potassium glutaminates (sic), formates, acetates, and lactates. Values for the amino compounds were higher than true dissolved solids, while values for the other salts were lower. Since the differences between true and refractometric solids as calculated to 100% dry substance varied for low and high concentration of the same non-sugar, Stanek hypothesized that on dilution there occurred, in addition to contraction, other physical changes such as dissociation, formation of hydrates, etc., which affected the refractive index.

Pulp (insoluble solids). The effect of pulp on refractometer values of fruit products has apparently not been the object of intensive study. Macara *(11)* treated it as only an insoluble diluent of jams. Stevens and Baier *(17)* remarked that insoluble solids were important, but reported no study of its effect on refractometer values. The National Canner's Association *Manual for the Canning Industry (20)* states that pulp in tomato paste does not affect the refractive index except to make the dividing line less distinct.

Peel oil. Refractive index is one of the characteristics commonly used to describe citrus oils. No reference has been found in the literature, however, on the effect of oil dissolved in fruit juices in the very low amounts found in citrus juice products.

Pectin. Kertesz *(10)* reported that the few values of refractive indives of commercial pectin extracts that had been published were of little value, as most of the extracts studied contained from 50 to 95% of solids other than pectic materials.

Flavonoids and ascorbic acid. No literature references have been found concerning the refractive indices of these components of citrus juices.

EXPERIMENTAL METHODS

Refractometer values. The instrument used in these studies was a Bausch and Lomb Precision refractometer [4] with refractive index range of 1.20 to 1.51. The scale is of the arbitrary equi-division type, with vernier scale attached to the alidade. Scale readings are converted to refractive indices and/or per cent sugar by use of tables accompanying the instrument. Temperature of the prisms was controlled by circulating water maintained at 20 ± 0.05° C. Temperature of the room did not exceed instrument temperature by more than 5° C. The refractometer was calibrated at about 35.7° Brix with 2,2,4-trimethylpentane and at about 51.5° Brix with methylcyclohexane, both certified by the National Bureau of Standards. These two solvents and double distilled water agreed to within one unit in the fifth decimal place. Calibration was effected by setting the crosshairs on the shadow line of trimethylpentane.

With clear test solutions and single strength juices, the shadow line is quite sharp. With concentrated sugar solutions, however, the shadow line becomes less distinct, and care must be used by the operator in setting the crosshairs at the proper point in the hazy shadow area. When concentrated citrus juices containing appreciable amounts of pulp are used, the shadow line becomes increasingly indistinct in direct proportion to the concentration. In order to avoid bias in setting the crosshairs, the practice was adopted of bringing the crosshairs into position alternately from each side with a minimum of 6 readings per sample.

It was found that with highly viscous concentrates best resolutions of the shadow line could be obtained by flowing the sample into the space between prisms by slight pumping action of the movable prism. Application of sample to the prisms with a rubber policeman, as is sometimes practiced with other refractometers, was not successful.

Minerals. The effect of the mineral content of juices on refractive index was studied by preparing solutions of potassium, magnesium, and calcium citrates and taking their refractive indices at various concentrations. According to data published by the Wisconsin Alumni Research Foundation in 1956 *(3)* potassium, magnesium and calcium occur in whole reconstituted orange concentrate at an average concentration of 187, 10.4, and 9.2 mg/100 g respectively. These proportions are of the same order of magnitude as those reported by a number of other investigators. If these minerals occur in the juices as citrates, the respective proportions of each metallic citrate can be calculated to be about 83 parts potassium citrate, 11 parts magnesium citrate, and 6 parts calcium citrate. The applicability of these calculations is supported by the analyses of orange juices, to be reported in another paper, in which the average potassium in the ash of 35 samples differed by less than 5% from the above proportion. Twenty-seven solutions containing these salts in approximately the above proportions and in concentrations ranging from 0.5 to 10% total salts were tested with the refractometer. Differences between actual solids contents and refractometer sugar scale values were plotted against actual solids contents to give a correction curve.

Amino compounds. Corrections for soluble nitrogenous components to be applied to refractometer sugar scale readings were studied by preparing a solution of amino acids in the proportions in which they were reported to occur in fresh Valencia orange juice by Underwood and Rockland *(21)*. The solution contained amino compounds in the proportions shown:

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Asparagine	0.31
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Serine	0.45
Aspartic acid	0.45
Proline	0.60
Arginine	0.60
Water to make	20.02 g

This solution was diluted with water to 1, 3, and 6% solids, and with sucrose syrup to 1.5 and 2.5% amino acid content, and tested with the refractometer. Differences between known solids and refractometer sugar scale values were plotted against the solids content, and a correction curve drawn.

Pulp (insoluble solids). The effect of pulp was studied by taking the refractometer sugar scale values of whole single strength juices, centrifuging, and taking the refractometer value of the supernatants. In other studies pulp was recovered by passing whole orange juice through a supercentrifuge equipped with a clarifier bowl. Different weights of pulp were added to sucrose syrups of known composition, and allowed to equilibrate at least as long as overnight. Meanwhile some of the recovered pulp was analyzed for moisture by vacuum drying, and the clarified juice tested for solids by the refractometer. Having knowledge of the composition of the juice with which the pulp was wet, and moisture content of the pulp, the amounts of soluble solids and water added to the syrup along with the pulp could be calculated. A typical calculation of the soluble solids content of a mixture of 8.00 g pulp containing 80% moisture (pulp wet with juice containing 10% soluble solids) and 105.00 g 40% sucrose syrup is as follows:

Moisture content of pulp (80% of 8)	6.40 g		
Moisture content of syrup (60% of 105)	63.00 g		
Total moisture in mixture		69.40 g	(61.416%)
Soluble solids in juice of pulp (10/90 of 6.40)	0.71 g		
Soluble solids content of syrup (40% of 105)	42.00 g		
Total soluble solids in mixture		42.71 g	(37.796%)
Dry pulp content of mixture (8.00–6.40–0.71)		0.89 g	(0.788%)
Grand Total		113.00 g	(100.00%)

By comparing calculated soluble solids contents of pulp and syrup mixture with their refractometer sugar scale values, corrections for the effect of pulp were obtained.

In further studies, pulp was recovered by centrifugation and washed with water; pulp was recovered by precipitation with alcohol, washed with acetone and dried; and pulp was recovered by centrifugation from juice of oranges damaged by freezing while still on the tree and from reconstituted frozen orange concentrate. In each instance, the pulp was added to sucrose syrups and changes in refractometer values noted over a period of several days.

Peel oil. Since citrus peel oils are very slightly soluble and consequently difficult to disperse uniformly in water, concentrated orange juice fresh from the evaporator, and consequently low in oil content, was selected as the medium for studying the effect of oil on refractometer values. Cold pressed orange oil was dispersed in the concentrate in amounts up to 0.5% by weight, which is about 10 times the amount considered optimum by most producers. Increases of sugar scale values above calculated soluble solids content were plotted against added oil.

Pectin. Commercial pectin was purified by dispersing in water, precipitating twice with acidified alcohol, washing free of acid, and redispersing in water. Anhydrogalacturonic acid (AGA) content of the solution was determined by the carbazole method. Portions of this 1.045% AGA solution were diluted with water, and their refractometer sugar scale values taken. Differences between refractometer values and actual solids contents as per cent AGA were plotted against the solids content. Other portions of the pectin solution were mixed with sucrose syrup and with solutions containing sucrose, citric acid, and potassium citrate. Differences between refractometer values and actual solids were plotted against AGA content of these mixed solutions, with corrections first being applied for the citric acid and potassium citrate present.

Ascorbic acid. High-purity l-ascorbic acid was added to approximately 50% sucrose syrup to give concentrations of 0.2% and 0.7%, and to water to give about 0.4, 0.5, 0.7, and 0.8% concentrations. Refractometer sugar scale values were determined and compared to calculated soluble solids content.

Flavonoids. Juice analyses indicate the presence of flavonoids in citrus juices in quantities far above their solubilities in water *(8, 12)*. Purified hesperidin is almost insoluble in water, while purified naringin has a solubility of only 0.05% in water at 20° C *(15)*. Since hesperidin and naringin are very similar in molecular composition, their refractive indices should be very nearly identical. Because of its solubility, naringin was selected as representative of citrus flavonoids for studying their effects on refractive index. Supersaturated solutions were prepared by dissolving naringin dihydrate in hot water, cooling, and diluting to several concentrations. Differences between refractometer sugar scale values and known solids contents of the solutions were plotted against solids contents.

Citric acid. The table of corrections for citric acid published by Stevens and Baier *(17)* in 1939 contains two sets of corrections, one for correcting refractometer readings to degrees Brix and the other to true soluble solids. The latter is of direct interest in this study. In order to check their corrections to soluble solids, solutions of citric acid were made up in water at intervals of 1% up to 10% concentration. Exact acid concentrations were determined by titrating weighed portions with standardized NaOH solution. Refractometer sugar scale values were taken at each concentration, and differences between these values and actual acid content were compared with the corrections of Stevens and Baier.

Reducing sugars. Refractive indices of glucose, fructose, and invert sugar solutions, as published in the 1955 *Official Methods of Analysis*, A.O.A.C. *(1)* were converted to sugar scale values by use of the International Scale of Refractive Indices of Sucrose Solutions *(1)* at concentrations up to 14% of the individual sugars and 28% invert sugar. The differences between these values and actual percentages of sugar are the amounts which must be added to refractometer sugar scale values to indicate true soluble solids.

RESULTS AND DISCUSSION

All components studied, other than citric acid and reducing sugars, were found either to have higher refraction than sucrose and thus to require negative corrections, or to be so nearly equal to sucrose that their effects were negligible.

The mineral citrates tested gave higher values on the refractometer sugar scale than the actual percentage of salts present, indicating the need to subtract corrections from the refractometer values. When differences between observed and actual solids contents of the mixed solutions were plotted against the solids contents, a smooth curve could be drawn which passed through the origin and missed no one of the 27 points by more than 0.02% solids. The list of corrections shown in Table 1 were taken from this curve.

For the solutions of mixed amino acids, the observed refractometer sugar scale values were higher

TABLE 1

Corrections for non-sugar components of concentrated citrus juices to be applied to refractometer sugar scale values to obtain true soluble solids

Per cent by wt	Citric acid[1]	Amino compounds	Mineral citrates	Pulp
	To add	To subtract		
0.2	.02	.05	.03	.02
0.4	.04	.10	.06	.05
0.6	.06	.16	.08	.10
0.8	.08	.21	.10	.17
1.0	.11	.26	.12	.25
1.2	.13	.32	.14	.35
1.4	.16	.37	.15	.46
1.6	.18	.43	.17	.58
1.8	.21	.48	.19	.70
2.0	.23	.53	.20	
2.2	.26	.59	.21	
2.4	.29	.64	.22	
2.6	.31	.69	.24	
2.8	.33	.74	.25	
3.0	.35	.80	.26	
3.2	.38	.85	.27	
3.4	.40	.90	.28	
4.0	.47			
5.0	.59			
6.0	.71			

[1] From Stevens and Baier (17).

than actual solids present, so negative corrections are required. When differences between observed and actual percentages were plotted against percentages of amino acids, the points lay on a straight line through the origin with only one point varying as much as 0.05% solids from the line. The list of corrections shown in Table 1 were taken from this curve.

The effect on refractive index of suspended matter, or pulp, has proven most difficult to measure and interpret. Tests indicated that changes in composition of single strength orange juice caused by centrifuging were not detectable with the refractometer. Obviously, the composition of the serum was not changed, but removal of the suspended solids and adsorbed material did change the over-all composition.

In the case of concentrated citrus juices, the ratio of pulp to solution is much greater than in single strength juice, and pulp should therefore exert considerable influence on physical characteristics, including refractive index, of concentrates. The addition of pulp to sugar syrup, or simulated concentrate, affects the refractive index considerably beyond the amount that can be attributed to mere dilution. Refractometer values were always higher than the calculated values. It appeared as if the insoluble solids had adsorbed some of the water from the syrup, leaving the solution more concentrated. This effect is not at variance with the observation that the removal of pulp with the supercentrifuge did not change the refractive index of single strength juices.

The condition of the pulp apparently influences the magnitude of its effect on refractive index. Differences were noted in the times required for mixtures to reach equilibrium with sucrose solution among pulp wet with juice, water washed pulp, and dried pulp, and also among final refractometer values of these pulp mixtures after allowing 5 or 6 days for equilibration. There was also noted a difference between the refractometer values of syrups with added pulp from frozen juice and from normal juice.

Pulp as recovered by centrifuging from citrus juices is a heterogeneous mixture which includes celluloses, protopectins, pectins, lipids, proteins, carotenoid pigments, and glycosides among other components. Some of these components adsorb water in varying amounts. Others, especially those of almost colloidal dimensions, scatter the light rays in the refractometer and render a determination of true refractive index difficult by obscuring the shadow line. Components occurring in discrete masses can also make determinations difficult by reducing the intensity of transmitted light and thus reducing the contrast between light and dark fields.

Characteristics of at least some of the components of pulp may be changed by freezing of the fruit, or by washing, precipitating or drying the experimentally recovered pulp, with the changes producing different effects on refractive index. The effects of such drastically treated pulps on refractive indices differed rather widely from those of pulp freshly recovered from normal juice. While effects of the latter were not always consistent, they did follow a trend. Figure 1 illustrates the effects of four different

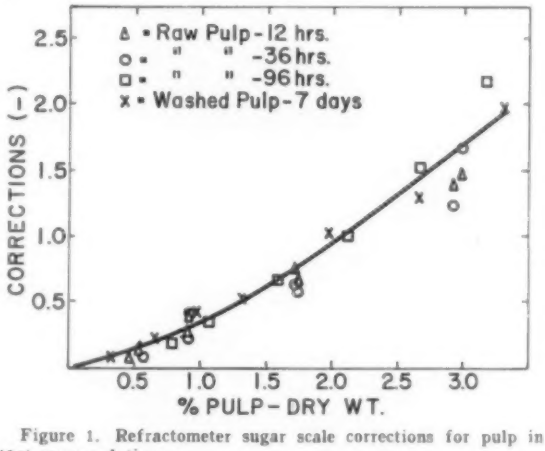

Figure 1. Refractometer sugar scale corrections for pulp in 40% sugar solution.

samples of pulp in 40% sucrose syrups. Differences between calculated soluble solids of mixtures containing pulp and observed sugar scale values were plotted as ordinates against known dry pulp content as abscissas for concentrations up to 3%. The list of corrections shown in Table 1 were taken from this curve. Pulp contents (dry weight) of 42° Brix concentrates seldom exceed 1.3%. Since the presence of pulp causes sugar scale values to read too high, the differences shown must be subtracted from refractometer values to indicate true soluble solids.

Citrus peel oil is a normal component of juice products and refractometer sugar scale values showed increases upon the addition of cold pressed orange oil to concentrated orange juice. In amounts normal to commercial concentrates, up to 0.04%, the differences were not larger than could be attributed to experimental error in sampling or reading the instru-

ment. When oil was added in larger amounts, up to 0.5% of the weight of concentrate, sugar scale increases were proportional to the amounts added. When these increases were plotted against oil concentration and extrapolated to zero oil content, the resulting curve indicated that corrections for concentrations normal to concentrated citrus juices amounted to less than 0.01° Brix. Corrections taken from this curve are shown in Table 2. Since peel oil in excess of 0.120% is not allowed under the Florida Citrus Code, its effect was considered negligible.

Differences between refractometer sugar scale values and pectin concentration, as per cent AGA, in water solution, when plotted against concentration, fell on a straight line passing through the origin. However, differences between sugar scale values and AGA concentration in 40% sugar syrup, with and without added citric acid and potassium citrate, showed random variations about a line with slightly lower slope than the one for pectin in water. The lack of precision in these measurements is not alarming when it is remembered that pectin does not go into true solution, but into colloidal dispersion. It is readily understandable that the hydrophyllic colloid would not disperse as readily in viscous syrups as in water. It is unlikely that complete uniformity of the sols was obtained. Differences between observed values and AGA concentrations are shown in Figure 2. Points are shown for pectin in water, pectin in

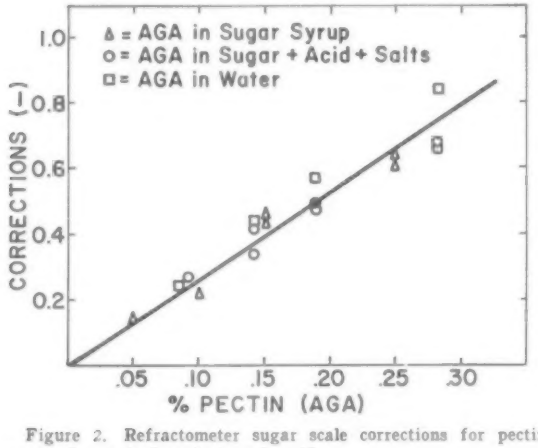

Figure 2. Refractometer sugar scale corrections for pectin measured as anhydrogalacturonic acid.

sugar syrup, and pectin in syrup containing acid and potassium citrate. Two points are shown at each concentration for the sugar solutions to indicate increases in values of the same solutions after standing for five days. The correction curve was drawn without regard for the pectin in water points, as the pectin in sugar solutions are considered to more closely resemble the situation existing in citrus juices and concentrates. Corrections taken from the curve are shown in Table 2 for pectin, as AGA, up to 0.35%.

The refractometer values of ascorbic acid in water solution at four concentrations were found to vary above and below those of equivalent sucrose values within the range of experimental error. When dissolved in 50% sugar syrup, ascorbic acid raised refractometer values only 0.01° Brix at 0.2% concentration, and 0.05° Brix at 0.73% concentration. Since this component seldom occurs in concentrated citrus at concentrations above 0.25%, its effect on refractometer values was considered negligible.

Flavonoid solutions gave values above true soluble solids contents, but increase with increasing concentration was very irregular. At the saturation point for naringin in water at 20° C, 0.05%, the refractometer reading was 0.17%, showing an increase of 0.12% above true soluble solids, while at concentrations (supersaturated) up to five times as great, the greatest increase was not more than 0.18%. Wide variations in increases at the various concentrations indicate that the use of supersaturated solutions was not entirely satisfactory, as naringin apparently seeded out of some of the solutions during the test. By plotting increases against soluble solids and fitting a curve to those points which did not indicate seeding, the corrections shown in Table 2 were developed.

TABLE 2

Corrections for non-sugar components of concentrated citrus juices to be applied to refractometer sugar scale values to obtain true soluble solids

Per cent by wt	Pectin	Flavonoids	Peel oil
	To subtract		
.02	.05	.07	.00
.04	.10	.10	.01
.06	.14	.12	.01
.08	.24	.14	.01
.10	.26	.15	.02
.15	.40	.17	.03
.20	.53	.18	
.25	.67	.18	
.30	.80		
.35	.93		

A satisfactory test for the routine estimation of flavonoids in orange juice has not been developed. The Davis test *(7)* involves the use of alkali which solubilizes flavonoids not in solution in the natural juice. Swift and Veldhuis *(19)* reported the presence, by the Davis test, of flavonoids in hand reamed and commercially extracted orange juices in concentrations very close to 0.03% throughout an entire season. Various investigators, using the Davis test, have reported the occasional presence in grapefruit juices of flavonoids in concentrations in excess of 0.05%, although the absence of excessive bitterness indicated that naringin was not present in such amounts. After careful consideration of available data, the authors concluded that flavonoids in single strength juices seldom exceed about 0.03%, and that, upon concentration of the juices, the flavonoids probably seed out upon reaching concentrations of about 0.06%. At this level, naringin causes an increase of about 0.12% in refractometer sugar scale values above true soluble solids. This amount may be used as a uniform negative correction for refractometer readings of all citrus concentrates.

Corrections for citric acid in solution agreed with those published by Stevens and Baier within 0.05%

solids. These differences were considered to be within experimental error. A partial listing of their corrections to true soluble solids is presented in Table 1, in order that they may be compared with corrections for other components occurring in similar concentrations.

Corrections for reducing sugars are shown in Table 3. The corrections for invert sugar, being slightly larger than the average of corrections for glucose and fructose of equivalent concentration, indicate the mutually depressing effect of the two reducing sugars. This effect amounts to about 0.03% sugar in the ranges in which these sugars occur in citrus juices. In most pure citrus juices, glucose and fructose are present in nearly equal quantities and the invert sugar corrections could be used. If the ratios deviate appreciably from 1:1, or glucose has been added, corrections for individual sugars may be preferable.

TABLE 3

Corrections for reducing sugars to be applied to refractometer sugar scale values to obtain true soluble solids[1]

Per cent by wt	Glucose	Fructose	Invert
	To add		
2	.013	.033	.033
4	.020	.053	.053
6	.020	.080	.073
8	.033	.107	.100
10	.053	.140	.123
12	.069	.163	.147
14	.094	.194	.169
16			.200
20			.261
24			.312
28			.343

[1] Data calculated from Tables 42.7 and 42.10, *Official Methods of Analysis*, A.O.A.C., 1955.

By inspection of the corrections for all the effective components of citrus juices in the range in which they are known to occur, it appears that the net correction to obtain true soluble solids should be negative. In other words, the refractometer sugar scale values as read, without corrections, are usually higher than actual soluble solids contents of the juice products, and the addition of corrections for citric acid only serves to compound the error, at least with low-acid products such as orange juice.

In the next paper of this series, the analyses of a number of commercial concentrates will be considered and corrections applied for the components in the proportions found.

SUMMARY

The effects of non-sucrose components of citrus juices on their refractometer sugar scale values have been studied. The only two components whose refractive indices in solution have been compared with sucrose and reported in the literature are citric acid and reducing sugars. Both of these components have lower indices of refraction than sucrose, and therefore give rise to a need for positive corrections for the refractometer sugar scale to indicate true soluble solids. Other components included in this study had higher indices of refraction than sucrose. Negative corrections required for both amino acids and pulp are greater than positive corrections required for citric acid at equivalent concentrations. Negative corrections required for citrate salts, flavonoids, and pectin are considerably less, while those for ascorbic acid and peel oil are practically negligible.

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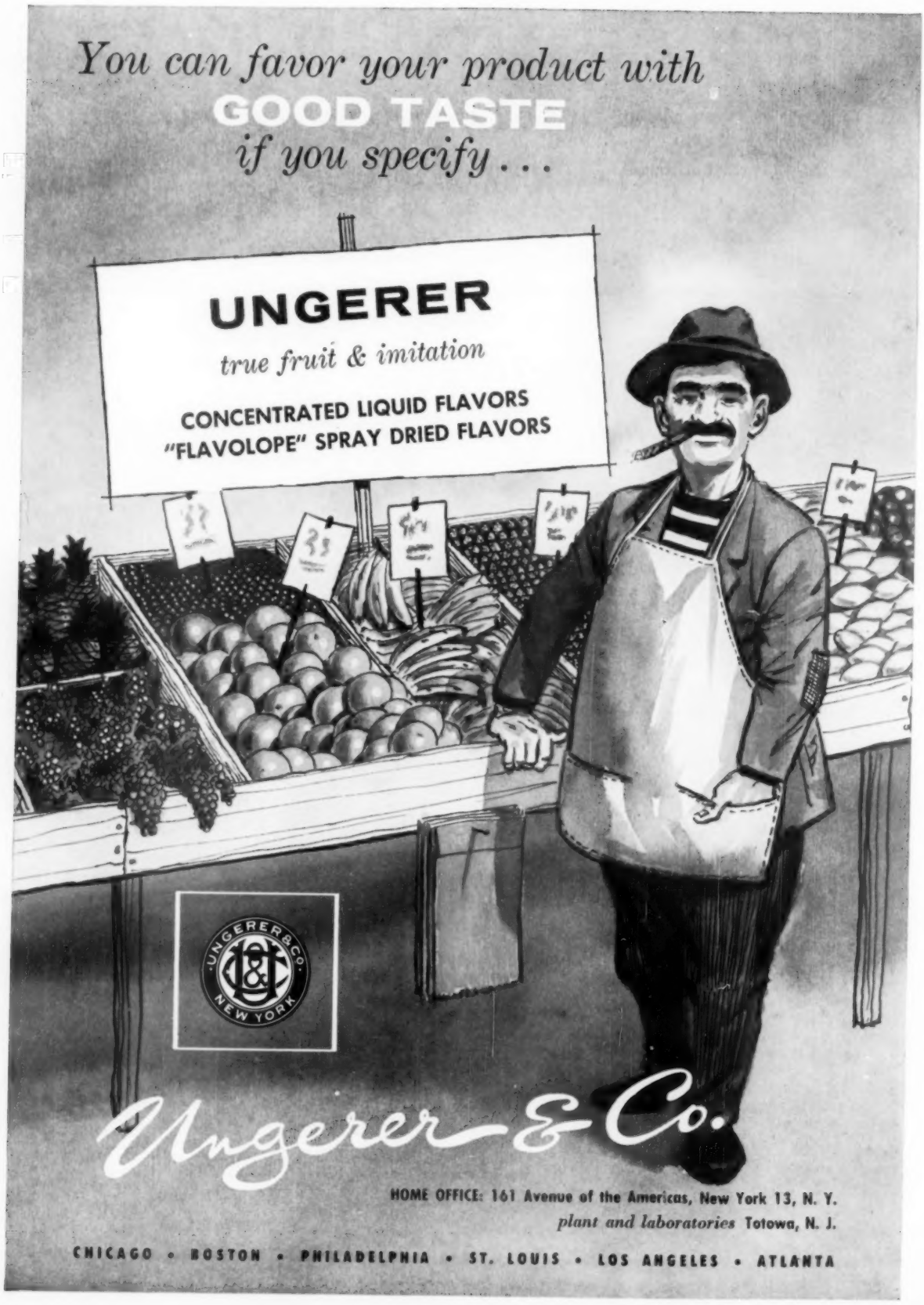

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Determination of the Proximate and Electrolyte Content of Beef

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(Manuscript received May 31, 1960)

RECENT STUDIES indicate that variations in meat composition, some so small as to appear superficial, may markedly affect meat quality. This is exemplified by the evidence that variation in proximate composition and ionic constituents, such as hydrogen and calcium, substantially influence the water retention of meat *(7, 16, 17)*. In meats, as in many other foods, acquiring a comprehensive knowledge of the relation between composition and quality offers promise of developing new ways in which quality may be improved.

It has long been apparent in this Laboratory that a system of meat analysis was needed which would permit the collection of a large amount of data in as short a time as possible. It was our opinion that a system could be developed, improving on available methods *(1, 6, 11)*, which would dispense with laborious and time-consuming separations, would use as far as possible a common ash solution for determination of electrolytes, and make extensive use of spectrophotometric methods. This paper describes such a system. It was successfully employed to collect the analytical data that has been reported *(16)* in a study on the variations in composition and properties among eight beef muscles.

MATERIALS AND METHODS

Preparation of samples. Muscle was removed from the carcass and trimmed of all visible fat and connective tissue. The muscle tissue was cut into approximately 1 in cubes and the cubes thoroughly mixed by shaking in a large polyethylene jar. One hundred grams of the meat were then blended to a fine paste in a Serval Omnimixer[a] which, because of its stainless steel construction, high speed, and cutting action, yielded a finely comminuted sample with a minimum of metallic contamination. Heating during blending was prevented by immersing the mixer jar (polyethylene) in an ice bath.

Determination of moisture. Into tared, high-form Vitreosil crucibles (50 ml size C) 15 ± 0.1 g portions of meat paste were weighed. The paste was spread to form a funnel shaped conduit to facilitate moisture loss. The crucibles were placed in a gravity convection oven, the temperature was slowly raised to 102–105° C, and the samples were held at this temperature overnight (16–18 hr) *(18)*. After cooling, the crucibles were weighed and the moisture content was calculated. The dried samples were retained for subsequent ashing and preparation of the stock electroyte solution.

Determination of fat. Content of fat was determined by a modified Babcock procedure. Nine grams of the original meat paste were weighed into 20% size, Paley-type Babcock bottles (Kimble Glass No. 508). Thirty ml of a 1:1 perchloric acid-acetic acid solution, prepared by mixing equal volumes of reagent grade perchloric acid of 60% strength and reagent grade glacial acetic acid, as recommended by Salwin *et al (13)*, were added. The contents were mixed by swirling, after which the bottles were immersed in a boiling water bath for 30–60 min. They were swirled occasionally in the bath. Satisfactory digestion was achieved when the meat solids had disintegrated into fine particles. The bottles were then removed, and additional acid mixture was added until the fat column rose into the calibrated neck of the bottles. The bottles were centrifuged for 2 min at 900 rpm in a head 15 in in diameter, and then placed in a 70° C water bath for 15 min. One half ml of the acid mixture was drained down the wall of the calibrated neck which momentarily separated the fat phase from the digest and permitted reading; otherwise, chromogens in the fat tended to obscure the fat-digest miniscus. From the amount of fat separated, the fat content as per cent wet weight of meat was calculated.

Determination of nitrogen. Nitrogen was determined by a micro-Kjeldahl procedure. Five grams of meat paste, plus 3 drops of octyl alcohol to prevent foaming, were blended with 100 ml of cold, ammonia-free water for 5 min in a Serval Omnimixer. Five ml of the slurry were transferred with a large bore pipette to a tared beaker and weighed. The amount of meat, in such an aliquot, was calculated, assuming the meat-water slurry to be homogeneous. Another 5 ml aliquot, containing approximately 8 mg of nitrogen, was placed in a 30 ml micro-Kjeldahl flask. Two ml of concentrated sulfuric acid, 2 drops of octyl alcohol, approximately 50 mg of a 1:1:3 mixture of mercuric oxide, potassium sulfate, and copper sulfate, and glass beads were added. The sample was gently boiled until frothing ceased, after which the sample was strongly heated until clear. After cooling, 15 ml of distilled water were added, and the solution, plus subsequent washings, was transferred to a steam distillation apparatus. Fifteen ml of 30% sodium hydroxide, plus 5% sodium thiosulfate, were added, and the alkaline mixture was distilled for 15 min. The ammonia was collected in a 125 ml Erlenmeyer flask receiver containing 5 ml of saturated boric acid solution and 2 drops of an indicator recommended by Ma and Zuayaga *(10)* (5 parts of 0.1% bromocresol green and 1 part of 0.1% methyl red, both in ethanol). The ammonia absorbed by the boric acid solution was titrated with 0.072 N sulfuric acid to a pink end point, 1 ml of the acid being equivalent to 1 mg of nitrogen. Protein was reported as N × 6.25.

Determination of chloride. The method of Kerr *(8)* for chloride in meat products was adapted. Ten grams of meat paste were weighed into a 125 ml Erlenmeyer flask. Fifteen ml of 0.02 N silver nitrate were added, followed by 10 ml of concentrated nitric acid. The flask was placed on a hot plate and heated at low heat until foaming and frothing ceased. Then 1 ml of saturated (5%) potassium permanganate was added, and the sample was heated for 15 min or until clear. Ten ml of water were then added and the solution brought to a boil. The solution was cooled to room temperature, and 20 ml of water were added. Prior to titrating, 1 ml of mononitrobenzene, as recommended by Caldwell *(5)* to prevent fading of the end point, 10 ml of ethyl ether to dissolve the fat, and 1 ml of 1 N nitric acid saturated with ferric ammonium sulfate were added to the solution. The solution was then titrated with 0.02 N ammonium thiocyanate to a pink end point, and the chloride content of the sample was reported as mg per cent wet weight of meat.

Preparation of meat ash. Ashing conditions were chosen which minimized contamination and volatilization of the elements, prevented fusion of the determinants with the silica crucibles, and produced a clear, colorless solution of the ash.

[a] Mention of specific commercial materials or equipment does not constitute recommendation for their use above similar materials and equipment of equal value.

The samples dried for the moisture determination described above were placed in a gravity convection oven, the temperature was slowly raised to 325° C, and the samples held at this temperature overnight to char. The samples were then placed in a Tempco muffle furnace (door closed), and the temperature was gradually raised to 400° C. At this stage, it was necessary to minimize air circulation, otherwise volatilizing fatty acids tend to ignite with possible loss of ash. After one hour at 400° C, the samples were cooled and the charred mass thoroughly wetted with 1 ml of concentrated nitric acid (prepared by distillation in an all-Pyrex, glass still). The samples were then placed on a hot plate and gently heated until the red-brown oxides of nitrogen were volatilized. The samples were replaced in the muffle furnace with the door slightly ajar to permit entrance of air. The temperature was raised to 525° C, and the samples were heated at this temperature for 2 hr. Some recalcitrant samples required two or three additions of nitric acid to produce the desired greenish-white ash. After cooling, the residues were weighed, and the ash was calculated and retained in the crucibles for analyses of electrolytes.

Preparation of electrolyte solution. Five ml of 0.1 N hydrochloric acid (prepared from all-Pyrex, glass-distilled acid) were added to each crucible. The solution of the ash was heated to boiling (to convert metal oxides to chlorides and pyrophosphate to orthophosphate) and then transferred to a 200 ml volumetric flask. Each crucible was washed repeatedly with 10 ml portions of hot, distilled water and the washings combined in the volumetric flask. The solution was adjusted to volume and transferred to polyethylene bottles.

Determination of phosphorus. Phosphorus present as the phosferrous orthophenanthroline complex *(4)*. The ml of the stock phosphoric acid complex *(9)*, which was stable for up to 2 days. Three ml of the stock electrolyte solution, containing approximately 150 meg/ml of phosphorus, were transferred to a Beckman absorption tube (diameter 25 mm, length 150 mm). Fifteen ml of 1.7 N nitric acid and 4 ml of an ammonium molybdate-ammonium meta vanadate mixed reagent were added. The mixed reagent consisted of a 1:1 mixture of 5% ammonium molybdate and 0.125% ammonium meta vanadate in 6 N nitric acid. The contents were thoroughly mixed and the absorbance of the molybdivanadophosphoric acid complex was determined at 470 mme in a Beckman B spectrophometer. The amount of phosphorus present in the aliquot was determined from a standard curve. Phosphorus content of the sample was reported as mg per cent wet weight of meat.

Determination of iron. Iron was determined as the orange ferrous orthophenanthroline complex *(4)*. Ten ml of the stock electrolyte solution, containing approximately 2 meg/ml of iron, were transferred to a Beckman absorption tube. Five ml of 10% hydroxylamine hydrochloride were added to convert ferric iron to the ferrous state. The solution was mixed and allowed to stand for 15 min; subsequently, 10 ml of 0.1% orthophenanthroline and, as recommended by Bandemer and Schaible *(2)*, 5 ml of 10% sodium citrate were added. The solutions were mixed and allowed to stand one hour for full color development. The absorbance of the orthophenanthroline-ferrous complex was determined at 520 mme in a Beckman B spectrophotometer. The amount of iron present in the aliquot was determined from a standard curve and reported as mg per cent wet weight of meat.

Determination of zinc. Zinc was determined as the zinc dithizonate complex in carbon tetrachloride solutions. Two ml of the common stock electrolyte solution, containing approximately 2 meg/ml of zinc, were transferred to a 100 ml polyethylene bottle. Fifteen ml of a mixed reagent, consisting of 10 parts water, 5 parts 2 N sodium acetate buffer pH 4.75, and 1 part 15% sodium thiosulfate, as recommended by Sandell *(14)*, were added. The contents were mixed, and 15 ml of 0.001% dithizone (in redistilled carbon tetrachloride) were added. The bottle was capped and vigorously shaken on a shaking machine for 15 min. The mixture was transferred to an absorption tube, the two phases were allowed to separate, and the absorbance of the carbon tetrachloride solution at 530 mme was determined. It was not necessary to separate the two phases, as the water phase was well above the light path of the Model B spectrophotometer. The amount of zinc present in the aliquot was determined from a standard curve; the zinc content of the sample was calculated as mg per cent wet weight of meat.

Determination of calcium. Calcium was precipitated as the oxalate by the method of Sendroy *(15)*, with subsequent oxidation of the oxalate by ammonium hexanitrato cerate as described by Berger *(3)*. The excess cerate was titrated with ferrous ion employing orthophenanthroline as an oxidation-reduction indicator. Fifty ml of the stock electrolyte solution, containing approximately 125 meg of calcium, were transferred to a 125 ml Erlenmeyer flask and slowly evaporated to dryness. Six ml of 1 N hydrochloric acid were added, and the solution was warmed. To 5 ml of this solution, in a 15 ml centrifuge tube, 2 ml of a 1:1 20% sodium acetate-saturated ammonium oxalate solution and 2 drops of bromocresol green indicator (0.1%) were added. The solution was adjusted to pH 4.5 and allowed to stand overnight to obtain complete precipitation. The tube was centrifuged at 3000 rpm for 15 min; the supernatant was decanted; and the precipitate was washed once with 2% ammonia and twice with a mixture of equal parts of ethyl alcohol, ethyl ether, and 2% ammonia. The precipitate was dried in an oven at 60° C. It was then dissolved by adding 2 ml of 2 N sulfuric acid and heating the mixture for 1 min in a boiling water bath. To the cooled solution, 2 ml of 0.014 N cerium ammonium nitrate were added. The solution was stirred and allowed to stand one hour. One drop of 0.005 M orthophenanthroline indicator was then added; and the excess cerate was titrated with 0.005 M ferrous ammonium sulfate, using a 5 ml microburette. The color change was from yellow to colorless, to a faint blue, and then to a sharp salmon-pink end point. The ferrous ammonium sulfate was standardized against 0.01 N sodium oxalate and the cerate solution against the iron solution. The amount of calcium present in the aliquot was determined from the titer. Total calcium content of the sample was reported as mg per cent wet weight of meat.

Determination of magnesium. The determination of magnesium was based on titrating with a standard solution of disodium ethylenediamine tetraacetate, employing a mixture of Eriochrome black T and methyl red as the indicator. Twenty-five ml of the common stock electrolyte solution, containing approximately 500 meg of magnesium, were transferred to a 500 ml Erlenmeyer flask and diluted to 275 ml with distilled water. Dilution of the sample avoided interaction of meat phosphate components with the Eriochrome black T and resulted in a sharper end point. Ten ml of a monoethanolamine hydrochloric acid buffer pH 10.3, as recommended by Patton and Reeder *(12)*, were added, together with 50 mg hydroxylamine hydrochloride and 100 mg potassium cyanide. The buffer solution consisted of 55 ml concentrated hydrochloric acid diluted to 400 ml, plus 310 ml redistilled monoethanolamine, the mixture diluted to 1 liter. Fifty mg of indicator, consisting of 0.2 g Eriochrome black T and 0.1 g methyl red triturated with 50 g of potassium chloride, were added. The solution was titrated with 0.02 N disodium ethylenediamine tetraacetate to a pale green end point. The disodium ethylenediamine tetraacetate solution was standardized by titrating separately, under similar conditions, against 500 meg of magnesium and 500 meg of calcium.

The calcium content of the 25 ml aliquot (determined separately as the oxalate) was subtracted from the titer, and the quantity of magnesium present was determined. The magnesium content of the sample was reported as mg per cent wet weight of meat.

Determination of sodium and potassium. Sodium and potassium were determined by flame photometry, employing a Beckman spectrophotometer equipped with a photomultiplier and a Model 9200 flame attachment. Interfering specific spectral emission was minimized by dilution of samples. Use of the photomultiplier permitted the selection of slit widths which minimized non-specific spectral emission.

Ten ml of the common stock electrolyte solution, containing 25–50 meg/ml of sodium and 200–300 meg/ml of potassium, were diluted to 50 ml. Sodium was determined at 589 mme, using photomultiplier tube (1P28); slit width, 0.02 mm; sensitivity, 0.1; photomultiplier position, 4; oxygen pressure, 10 lb;

TABLE 1

Eighteen analyses on a meat paste

Replicates[1]	% Wet weight of meat				Mg/100 g wet weight of meat							
	Moisture	Ash	Protein (N x 6.25)	Fat	Chloride	Magnesium	Calcium	Zinc	Sodium	Phosphorus	Potassium	Iron
1	72.95	1.18	23.00	2.0	40.2	23.6	3.38	3.84	42.6	197.1	407.0	2.24
2	72.95	1.20	23.21	2.0	41.2	23.3	3.38	3.61	46.4	197.3	419.5	2.20
3	72.68	1.22	22.97	2.2	41.2	24.4	3.37	3.98	42.5	198.9	416.0	2.28
4	72.89	1.17	22.80	2.0	40.0	22.9	3.39	3.98	46.5	197.4	425.9	2.23
5	72.73	1.19	22.94	2.1	40.3	23.1	3.36	3.84	41.6	197.3	420.8	2.17
6	71.50	1.22	23.32	1.9	39.8	22.9	3.39	3.98	46.8	197.4	421.4	2.17
7	72.82	1.23	23.15	2.0	39.5	23.0	3.38	3.99	41.6	199.4	414.7	2.31
8	72.62	1.19	23.06	2.0	41.3	23.4	3.38	3.83	43.7	202.1	420.7	2.29
9	72.70	1.13	23.23	2.2	39.6	23.6	3.35	3.84	42.6	199.8	420.1	2.24
10	72.99	1.21	23.29	2.1	36.0	23.5	3.35	4.10	42.4	199.7	417.9	2.29
11	72.96	1.24	23.00	2.2	40.4	23.2	3.39	3.83	46.7	199.4	426.0	2.23
12	72.72	1.21	23.23	1.9	40.3	23.6	3.38	4.08	44.8	196.1	422.0	2.23
13	72.24	1.24	23.02	2.2	40.1	23.3	3.32	3.84	45.2	201.1	425.0	2.24
14	71.31	1.22	22.97	2.0	40.7	24.4	3.41	4.06	45.1	197.9	425.5	2.24
15	71.93	1.29	23.21	2.1	40.6	23.9	3.39	3.96	46.6	200.0	423.8	2.29
16	71.93	1.21	22.91	1.9	40.6	24.3	3.36	4.00	46.6	202.6	423.4	2.29
17	71.56	1.23	23.23	2.0	40.0	23.6	3.34	3.95	42.8	197.6	427.4	2.37
18	69.58	1.18	23.38	2.2	39.5	23.5	3.34	3.71	45.3	200.1	422.8	2.38
Average value	72.28	1.21	23.11	2.1	40.1	23.5	3.37	3.91	44.4	198.9	421.1	2.26
Coef. of variation	1.21%	2.94%	0.69%	5.62%	2.89%	2.05%	0.68%	3.15%	4.21%	0.92%	1.18%	0.82%

[1] Analyses were made on eighteen 15-g samples, except that for the protein and chloride determinations separate samples, 5 and 10 g, respectively, were used.

TABLE 2

Recovery of ions added to meat

Aliquot	Mg/100 g wet weight							
	Chloride	Magnesium	Calcium	Zinc	Sodium	Phosphorus	Potassium	Iron
1	114.6	40.36	6.73	6.95	82.8	363.2	804.6	5.73
2	114.6	40.74	6.49	6.95	70.4	364.6	815.0	5.81
3	114.4	41.84	6.68	7.19	77.8	364.9	827.0	5.65
4	116.9	40.52	6.75	7.22	77.6	366.5	827.4	5.60
5	115.5	40.62	6.60	7.00	73.4	363.2	819.0	5.61
6	116.6	39.96	6.54	7.15	77.2	362.7	819.4	5.59
Average	115.5	40.7	6.64	7.08	76.6	364.2	818.6	5.67
Amount in meat	401.1	23.5	3.37	3.91	44.4	198.9	421.1	2.26
Amount added	71.0	16.7	3.33	3.33	33.3	166.6	376.3	3.33
Per cent recovery	106%	103%	97%	94%	97%	99%	103%	102%

and acetylene pressure, 3.5 lb. To compensate for the enhancement of radiation due to the potassium present in meat samples, 60 meg of potassium were incorporated in each ml of the sodium standards.

Potassium was determined at 768 mmc, using a cesium oxide, red sensitive phototube, slit width, 0.25 mm; sensitivity, 0.2; resistor, 2000 ohms; oxygen pressure, 10 lb; and acetylene pressure, 3.5 lb. At the level of sodium present, approximately 10 ppm, interference from this ion could not be detected in determining potassium.

RECOVERY EXPERIMENTS

To a residue, prepared by drying a 15 g sample of meat paste, a solution was added containing amounts of magnesium, calcium, zinc, sodium, phosphorus, and iron approximately equivalent to that in tissue. The solution was slowly evaporated to dryness, and the residue was charred and then ashed. The ash was dissolved in 0.1 N hydrochloric acid and diluted to 400 ml. Subsequent analysis for each electrolyte was performed as previously described.

To a 10 g sample of meat paste, 10 ml of 0.02 N sodium chloride were added and thoroughly mixed. Thirty ml of 0.02 N silver nitrate were added, followed by 10 ml of concentrated nitric acid, and the sample was analyzed for chloride as described.

ANALYTICAL RESULTS

Table 1 gives the results of the analysis of 18 replicate analyses of the same meat paste. Quantitative results are given for 12 constituents. The coefficient of variation of the determinations ranged from 0.68% to 5.6%.

Table 2 gives the results of the recovery of 6 replicate analyses of the same meat paste. The percentage recovery ranged from 94% to 106%.

SUMMARY

A convenient plan of analysis for the determination of the proximate and major electrolyte content of beef has been described. With this procedure, the coefficient of variation for the constituents was as follows: moisture, 1.21%; ash, 2.94%; protein, 0.69%; fat, 5.62%; chloride, 2.89%; magnesium, 2.05%; calcium, 0.68%; zinc, 3.15%; sodium, 4.21%; phosphorus, 0.92%; potassium, 1.18%; and iron, 2.26%. The average recovery of various ions was as follows: chloride, 106%; magnesium, 103%; calcium, 97%; zinc, 94%; sodium, 97%; phosphorus, 99%; potassium, 103%; and iron, 102%.

Acknowledgment

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432A

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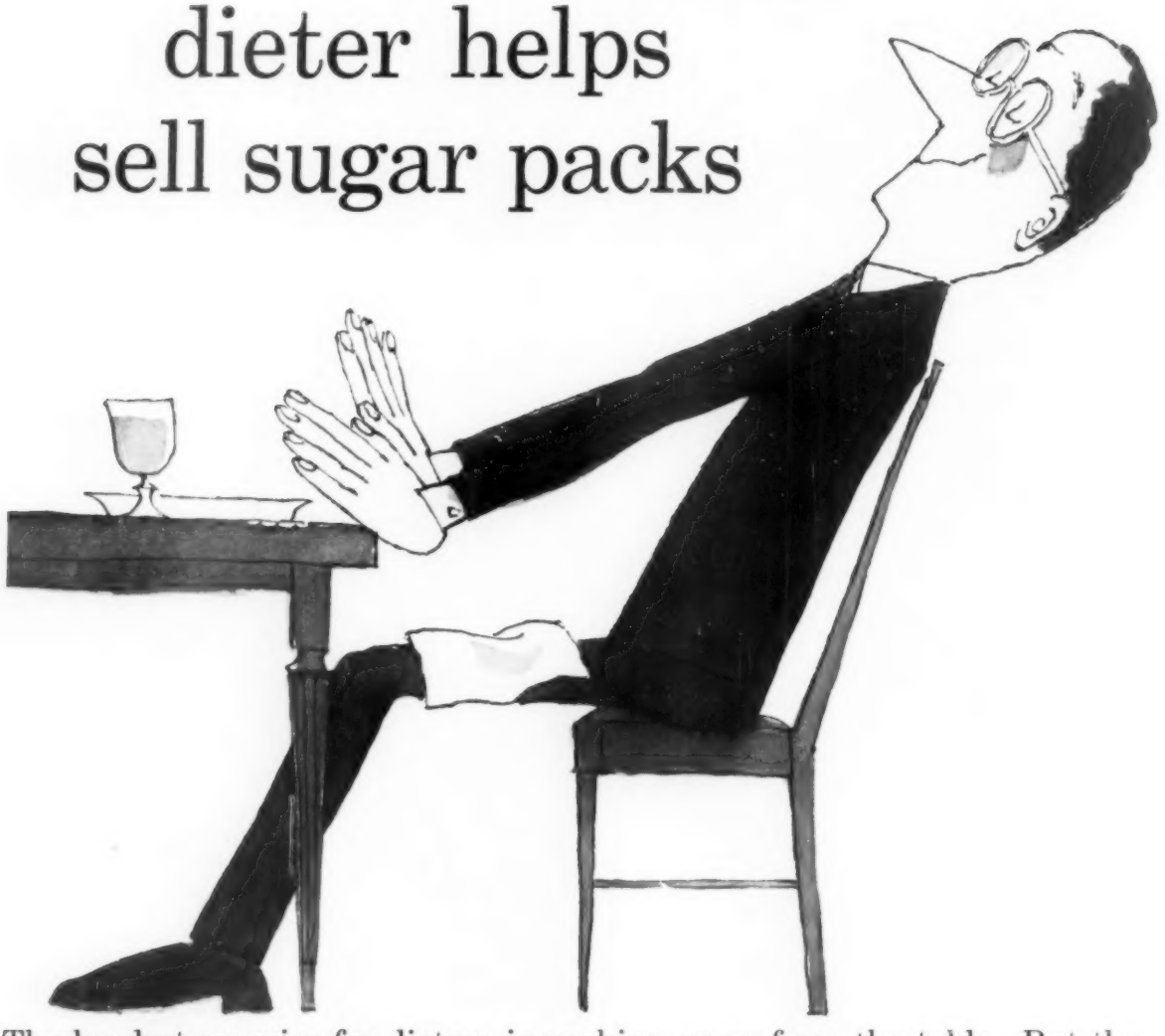

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432B

Effect of Sodium Chloride, Citric Acid, and Sucrose on pH and Palatability of Canned Tomatoes*

(Manuscript received May 10, 1960)

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TOMATO ACIDITY has been of particular interest to many investigators because it is related to ripeness, flavor, and processing requirements for canning *(1, 3, 6, 10, 12)*. The high pH values in fresh tomatoes during the 1957 and 1958 seasons were a matter of concern to the canning industry since an increased incidence of spoilage, and even a public health problem may develop when the pH value rises above 4.5 *(6)*. Higher temperatures and longer processing times could be used to assure the destruction of bacterial spores. However, this requires cooking under pressure and causes a decrease in color, flavor, and wholeness of the canned product. One solution is addition of citric acid to decrease the pH and thus to facilitate the heat sterilization of canned whole tomatoes at 212° F.

Kertesz *et al (5)* and Siegel *(9)* recommended the use of calcium in the commercial canning of whole tomatoes. The calcium-treated tomatoes yielded a higher drained weight and showed much more resistance to conditions causing disintegration than fruit canned in the usual manner. However, no information is available on the effect of citric acid acidification on drained weight of the canned product.

In the present experiment, the effect of ripeness level on pH and acidity of five varieties of tomatoes was studied. Presented are the effects of sodium chloride, citric acid, and sucrose on pH, palatability, and drained weight of canned whole tomatoes.

EXPERIMENTAL MATERIALS AND METHODS

Tomatoes. Two-hundred-pound lots of commercial tomato samples, which passed the California Department of Agriculture grading station, were canned for the varietal study. The raw fruit used for determining the effect of ripeness level on pH and acidity was obtained from commercial fields where tomatoes of 3 different ripeness levels were picked the same day on a basis of skin color.

Canning. Well-colored tomatoes, sorted under a standard light source, were used for canning. Unusually large and small fruits were removed. Tomatoes were lye peeled with a 5% sodium hydroxide spray at 218° F. Radiant heat and steam were applied in the peeler. The fruit was washed with a water spray, cored in a Magnuson "Hydrout" Model FF unit, and filled into No. 2½ cans with an average fill-weight of 21.5 ± 0.5 oz. Four oz of tomato juice, containing varying amounts of citric acid, sucrose, and NaCl, and enough $CaCl_2$ to make 0.07% of the whole content of the can, was added by a Magnuson "Flocron" Model E-3 dispenser. Tomato juice was then added to make a net weight of 28 oz for each No. 2½ can. The juice was prepared from tomatoes which were heated rapidly in a steam-jacketed kettle at 212° F and passed through a "Ryder" juice extractor with a 0.027 in screen. Cans were sealed under 16 in of vacuum, cooked for 32 min at 212° F in a rotary cooker, cooled for 7 min in a rotary water-cooler, then stored at room temperature for 2 to 3 months prior to chemical and flavor evaluation.

Color. Color of the tomato halves was measured with the Agtron Model E *(11)* which measured the ratio of green (546 mme) to red (640 mme) reflectance. An average value from 20 separate readings was reported for each sample. According to the standards of the California Department of Agriculture, the Agtron E readings for tomato color grades are as follows: well-colored, 0 to 46; fairly well-colored, 47 to 67; and off-color, 68 or higher.

Chemical analyses. Twenty representative tomatoes were taken from the fresh fruit and analyzed for pH, Brix, and titratable acidity. Twelve cans of the tomatoes were analyzed 3 months after canning. The pH was measured with a Beckman glass electrode pH meter. Soluble solids content was measured with a Zeiss-Opton refractometer, and the readings were corrected to 20° C. Titratable acidity was determined with a Beckman titrator. Duplicate samples of 10 g of tomato pulp were treated in 150 ml of distilled water with 0.1 N sodium hydroxide to pH 8.0. Results were reported as per cent citric acid. Salt in the canned tomatoes was determined by the $AgNO_3$ titration method with potassium chromate as an indicator *(2)*. Results were reported as per cent sodium chloride.

Drained weight. Drained weight of the canned tomatoes was determined according to U.S.D.A. procedure by draining the tomatoes for 2 min on a standard screen. The average drained weight of 12 cans was used for evaluation. Per cent retention was calculated as 100 times the ratio of drained weight to fill weight.

Flavor evaluation. A trained, laboratory taste panel of 14 members evaluated the flavor quality of the canned tomatoes over a period of 4 weeks. To facilitate testing, the samples were subdivided into 3 groups:

Group 1. Salt variations: Samples 1 through 6.

Group 2. Sucrose and acid variations: Samples 7 through 19.

Group 3. Varietal variations: Samples 20 through 34.

Using a balanced, incomplete block design *(4)*, 3 samples were judged per session for Groups 1 and 3, whereas 4 samples were judged per session for Group 2. The tomatoes were ranked 1st, 2nd, and 3rd in acceptability, as well as being scored on a scale with "0" denoting an inedible sample and "8" indicating excellent flavor quality. A total of 25, 20, and 21 separate flavor evaluations were obtained for each sample within Groups 1, 2, and 3, respectively. Both the ranks and the scores were subjected to analysis of variance for determination of significance.

RESULTS

Color and pH of fresh tomatoes. The relationship between color and pH of 5 varieties of California tomatoes is shown in Table 1. Cross-sectional areas of tomato halves were graded

TABLE 1

Influence of ripeness level on pH of fresh tomatoes* (1959 crop)

Variety	Under-ripe		Canning-ripe		Soft-ripe	
	Agtron E	pH	Agtron E	pH	Agtron E	pH
Pearson	68	4.20	38	4.50	36	4.60
Pearson VF6	71	4.30	33	4.50	30	4.70
Pearson VF11	54	4.14	49	4.30	36	4.50
CPC No 2	65	4.50	47	4.50	36	4.60
Ace	71	4.50	44	4.60	35	4.80
Average	66	4.33	42	4.48	35	4.64

* L.S.D. (pH difference due to ripeness level)
= 0.04 at 95% probability level
= 0.05 at 99% probability level

* Presented in part at the Twentieth Annual Meeting of IFT, San Francisco, California, May 18, 1960.

TABLE 2

Influence of ripeness level on soluble solids and titratable acidity of fresh tomatoes (1959 crop)

Variety	Under-ripe			Canning-ripe			Soft-ripe		
	Brix at 20° C	Titrable acidity, %	Brix/Acid	Brix at 20° C	Titrable acidity, %	Brix/Acid	Brix at 20° C	Titrable acidity, %	Brix/Acid
Pearson	6.19	0.481	12.9	6.09	0.359	17.0	5.79	0.322	18.0
Pearson VF6	5.85	0.441	13.3	5.85	0.333	17.6	6.05	0.204	29.7
Pearson VF11	5.35	0.342	15.6	5.75	0.329	17.5	5.45	0.263	20.7
CPC No 2	4.75	0.362	13.1	4.95	0.277	17.9	4.95	0.240	20.6
Ace	5.65	0.412	13.7	5.76	0.263	21.9	5.95	0.217	27.4
Average	5.56	0.408	13.7	5.68	0.312	18.4	5.64	0.249	23.3

L.S.D.: Acidity due to ripeness level = 0.020 at a 99% probability level.

for color by the Agtron Model E. As the tomatoes ripened, increase in red reflectance and decrease in green reflectance caused a decrease in Agtron E readings. Average readings for the 5 varieties decreased from 66 in under-ripe to 42 in canning-ripe, and to 35 in soft-ripe tomatoes. The average pH value increased from 4.33 in under-ripe to 4.48 in canning-ripe, and to 4.64 in soft-ripe fruit. These results are significant at the 99% probability level.

Soluble solids and titratable acidity. Soluble solids and titratable acidity of 5 tomato varieties at under-ripe, canning-ripe, and soft-ripe stages are shown in Table 2. No clear-cut trend was observed regarding the effect of ripeness level on soluble solids content. However, as the fruit ripened, titratable acidity decreased from 0.408% in under-ripe, to 0.312% in canning-ripe, and to 0.249% in soft-ripe fruit. These results are significant at the 99% probability level. Ripeness level could also be compared on the basis of the Brix/acid ratio which increased as the tomatoes ripened; that of soft-ripe fruit was 18 to 20 or higher, whereas that of under-ripe fruit was 12 to 15.

Effect of salt concentration on flavor. The effect of salt concentration on chemical and flavor analyses of canned whole tomatoes is shown in Table 3. The pH values of the unacidified fruit (Samples 1, 2, and 3) were 4.18 to 4.30, while those of the samples acidified with 0.3% citric acid (Samples 4, 5, and 6) varied from 3.92 to 3.95. Flavor evaluation of the canned product indicated that even though Sample 2 (0.75% salt) scored higher than Sample 1 (0.5% salt) and Sample 3 (1.0% salt), the differences were not significant. No significant differences in flavor were observed between Samples 4, 5, and 6, but there was a trend toward preference for Sample 6.

TABLE 3

Effect of salt concentration on chemical and flavor analyses of canned CPC No 2 tomatoes

Sample No	1	2	3	4	5	6
Added NaCl (%)	0.50	0.75	1.00	0.50	0.75	1.00
Added sucrose (%)	0	0	0	3.00	3.00	3.00
Added citric (%)	0	0	0	0.30	0.30	0.30
Chemical analysis						
% Salt as NaCl	0.63	0.88	1.07	0.61	0.78	0.93
% Total acidity as citric	0.367	0.368	0.384	0.591	0.600	0.586
pH	4.30	4.30	4.18	3.92	3.95	3.92
Soluble solids, Brix at 20° C	6.89	7.28	7.39	9.50	9.84	9.63
Brix/acid ratio	18.9	19.9	19.3	16.1	16.5	16.5
Flavor analysis[1]						
Average score	4.28	4.84	4.24	4.28	4.04	4.64
Average rank	2.16	1.72	2.04	2.20	2.08	1.80

[1] Average of 25 separate evaluations. No significant differences in scores or in ranks.

Effect of citric acid and sucrose on flavor. Table 4 summarizes the effect of citric acid and sucrose on the chemical and flavor analyses of canned tomatoes of the CPC No 2 variety. Sample 13, which contained 1.0% salt, 2.0% sucrose, and 0.2% citric acid was scored higher in flavor than Samples 9 and 12. In general, the addition of citric acid impaired palatability, whereas a combination of citric acid and sucrose improved it. For each 0.1% of citric acid added, 1.0% sucrose should be added to compensate for the sour taste. This is illustrated by comparing the flavor scores of Sample 10 with 11, 12 with 13, 14 with 15, and 16 with 17 and 18. There is no apparent advantage in using more than 0.2% citric acid and 2.0% sucrose.

Varietal differences. The effect of citric acid and sucrose on chemical and flavor analyses of 5 varieties of canned tomatoes is shown in Table 5. Results are similar to those in Table 4, i.e., acidification with citric acid impaired the palatability, while a combination of citric acid and sucrose improved it. The Ace variety was the least acceptable while the Pearson VF-6 was the most acceptable. Table 6 shows the analyses of variance for flavor scores and ranks.

Drained weight. The effect of citric acid and sucrose on drained weight of canned tomatoes is shown in Table 7. Acidification with 0.1% citric acid resulted in a slight decrease in drained weight. The per cent retention in the control sample (A) was slightly higher than in the acidified samples (B, D, F, H). Sucrose in excess of 1% of the filled weight resulted in a slightly lower drained weight.

DISCUSSION

Results obtained in this investigation indicate that ripeness and variety are important factors related to high pH in tomatoes. In order to obtain a high-quality product, harvest maturity should be more carefully considered. Ripe tomatoes should not be left on the vine for a long period of time, even though State grading emphasizes full, red color for fresh tomatoes, leading growers to harvest soft-ripe fruit. Also, if delay occurs between delivery and processing,

TABLE 4

Effect of citric acid and sucrose concentration on chemical and flavor analyses of canned tomatoes—CPC No. 2 variety

Sample No	7	8	9	10	11	12	13	14	15	16	17	18	19
Added NaCl, %	0.5	0.75	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
Added sucrose, %	0	0	0	0	1.0	0	2.0	0	3.0	0	2.0	4.0	6.0
Added citric, %	0	0	0	0.1	0.1	0.2	0.2	0.3	0.3	0.4	0.4	0.4	0.4
Chemical analysis													
% Salt as NaCl	0.63	0.88	1.12	1.05	1.05	1.10	1.05	1.12	0.88	1.07	0.93	0.98	0.85
% Total acidity as citric	0.396	0.368	0.368	0.473	0.446	0.532	0.508	0.617	0.681	0.727	0.667	0.649	0.635
pH	4.19	4.30	4.24	3.98	4.10	3.94	3.85	3.80	3.79	3.74	3.74	3.80	3.82
Brix at 20° C	7.41	7.28	7.65	7.76	8.72	7.73	9.31	7.90	9.99	7.91	9.49	10.45	11.36
Brix/Acid	18.8	19.9	20.8	16.5	19.6	14.6	18.4	12.8	14.7	10.9	14.2	16.1	17.9
Flavor analysis[1]													
Average score	4.65	4.45	4.10	4.60	5.20	4.05	5.10	3.75	4.10	2.55	4.00	4.25	3.40
Average rank	2.25	2.05	2.50	2.20	1.85	2.75	1.80	3.05	2.60	3.45	2.55	2.60	2.85

[1] Average of 20 separate evaluations. L.S.D. at the 5% level = 1.11 for scores and 0.63 for ranks.

TABLE 5

Effect of citric acid and sucrose on chemical and flavor analyses of canned tomatoes

Variety	Pearson			Pearson VF-6			CPC No. 2			Ace			VF-11		
Sample No	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34
Added NaCl, %	0.5	1.0	1.0	0.5	1.0	1.0	0.5	1.0	1.0	0.5	1.0	1.0	0.5	1.0	1.0
Added sucrose, %	0	0	3.0	0	0	3.0	0	0	3.0	0	0	3.0	0	0	3.0
Added citric, %	0	0.3	0.3	0	0.3	0	0	0.3	0.3	0	0.3	0.3	0	0.3	0.3
Chemical															
% Salt[1]	0.68	1.22	1.05	0.75	1.15	1.02	0.70	1.17	0.98	0.66	1.27	1.10	0.68	1.12	1.00
% Total acidity[2]	0.382	0.628	0.580	0.438	0.693	0.414	0.316	0.647	0.527	0.328	0.489	0.547	0.336	0.531	0.586
pH	4.24	3.81	3.83	4.27	3.98	4.30	4.33	3.84	3.88	4.28	3.98	3.90	4.23	3.80	3.92
°Brix	6.92	7.50	9.63	6.48	7.31	9.47	6.39	7.65	9.49	5.87	7.03	9.08	6.39	7.02	9.63
Brix/Acid	18.2	12.0	16.6	14.8	10.6	23.0	20.3	11.8	18.1	18.0	14.4	16.7	19.4	13.3	16.5
Flavor[3]															
Av score	3.86	3.95	4.90	4.14	4.10	4.67	3.43	3.14	3.71	3.29	2.81	3.67	3.43	3.19	4.10
Av rank	1.90	2.10	1.67	1.90	2.00	1.57	2.10	2.33	1.81	1.86	2.48	2.14	2.33	2.14	1.67
Variety av score		4.24			4.30			3.43			3.25			3.57	

[1] Calc. as NaCl.
[2] Calc. as citric acid.
[3] Average of 21 separate evaluations. For scores, treatment L.S.D. at 5% level = 1.17; Variety L.S.D. at 5% level = 0.96.

and the temperature is favorable to post-harvest ripening, tomatoes reaching the canning line may have pH values of 4.5 or higher. Proper control of these two factors would help to solve the pH problem.

Anderson *(1)* has investigated the factors affecting acidic constituents in tomatoes and has indicated that the temperature at which the fruits were ripened did affect the development of acidity. The occurrence of seasonal trends in acidity were inconsistent, and level of acidity varied for different seasons and years. It may be inferred that the high pH occurring in the 1957 and 1958 seasons was due to weather conditions or delayed harvest. If such conditions should occur again, it would be desirable to acidify the fruit with citric acid during processing.

Immediately after processing, the pH of the canned tomatoes was about the same as that of the fresh fruit. After storage for 2–3 months at room temperature, the pH of the canned product was 0.2 to 0.3 units lower than that of the fresh fruit. This might be attributed to (a) the hydronium ion derived from hydrolysis of calcium chloride; (b) formation of organic acids from degradation of reducing sugars; (c) carbon dioxide formation from the browning reaction between sugars and amino acids; or (d) other unknown causes.

Kertesz *et al (5)* reported that treatment of canned whole tomatoes with calcium chloride improved the firmness of the fruit and increased the number of tomatoes which remained whole in the can. The resistance of canned tomatoes to physical damage normally occurring during shipping was increased. In the present investigation, acidification with 0.1 to 0.4% citric acid caused a slightly lower drained weight. Retention was 93% in the unacidified sample and 88–89% in the acidified samples. This difference could be attributed, in part, to the difference in salt

TABLE 6

Analyses of variance

Group 1. Effect of salt concentration (Table 3)

Source of variation	Degrees of freedom	Flavor scores		Flavor ranks	
		Mean square	F ratio	Mean square	F ratio
Total	149				
Treatment	5	2.17	0.82	0.96	1.55
Remainder	144	2.66		0.62	

Group 2. Effect of sucrose and citric acid concentration (Table 4)

Total	259				
Treatment	12	9.79	3.05***	3.84	3.49***
Remainder	247	3.21		1.10	

L.S.D. at 5% level = 1.11 (scores)
L.S.D. at 5% level = 0.63 (ranks)

Group 3. Effect of varietal differences (Table 5)

Total	314				
Treatment	14	6.92	1.85*	1.48	2.39**
Variety	4	14.54	3.88**	1.21	1.95
Remainder	296	3.75		0.62	

Treatment L.S.D. at 5% level = 1.17.
Variety L.S.D. at 5% level = 0.96.

* Significant at 95% level of probability.
** Significant at 99% level of probability.
*** Significant at 99.9% level of probability.

TABLE 7

Effect of citric acid and sucrose on drained weight of canned whole tomatoes (CPC No 2 variety, 1959 pack)

Sample	Citric acid added, % of fill wt	Sucrose added, % of fill wt	NaCl added, % of fill wt	Average fill weight, oz	Average drain weight, oz (2 month cutout)	Per cent retention,* $\frac{\text{Drained wt}}{\text{Fill wt}} \times 100$
A	0.0	0.0	0.5	21.4	19.9	93.0
A1	0.0	0.0	1.0	21.5	19.7	91.5
B	0.1	0.0	1.0	21.5	19.3	89.6
C	0.1	1.0	1.0	21.4	19.3	90.0
D	0.2	0.0	1.0	20.8	19.1	91.8
E	0.2	2.0	1.0	21.1	18.4	87.1
F	0.3	0.0	1.0	21.6	19.3	89.3
G	0.3	3.0	1.0	21.5	19.1	88.9
H	0.4	0.0	1.0	21.5	19.1	88.8
I	0.4	2.0	1.0	21.6	19.8	91.5
J	0.4	4.0	1.0	21.5	19.2	89.3
K	0.4	6.0	1.0	21.5	18.6	86.5

* L.S.D. (Per cent retention): 2.8 at 95% probability level; 3.6 at 99% probability level.

concentration between the control sample (0.5% salt) and the acidified samples (1.0% salt). Siegel *(8)* presented evidence that the addition of table salt to canned tomatoes caused softening of the tissues and results in a slightly lower drained weight. Kertesz *et al (5)*, indicated that the drained weight increased from 74.8% retention in the control to 83.6% in the samples treated with 0.067% calcium chloride. It is apparent from the present investigation that acid and sugar additions cause only minor changes in drained weight of the canned product.

SUMMARY

Selected lots of California tomatoes were acidified with citric acid to lower the pH and to facilitate heat sterilization. Varying amounts of sodium chloride and sucrose were added to tomatoes of five varieties. Three months after canning, the tomatoes were analyzed for pH, acidity, soluble solids, drained weight, and flavor acceptability. Addition of citric acid effectively lowered pH but impaired palatability. The proper combination of citric acid, sucrose, and sodium chloride improved flavor quality over untreated controls within each variety. The Agtron E color readings decreased from 66 in under-ripe to 42 in canning-ripe, and to 35 in soft-ripe tomatoes. Average pH values increased from 4.33 in under-ripe to 4.48 in canning-ripe, and to 4.64 in soft-ripe fruit. Changes in pH during storage of the canned product for 3 months at room temperature are discussed.

Acknowledgments

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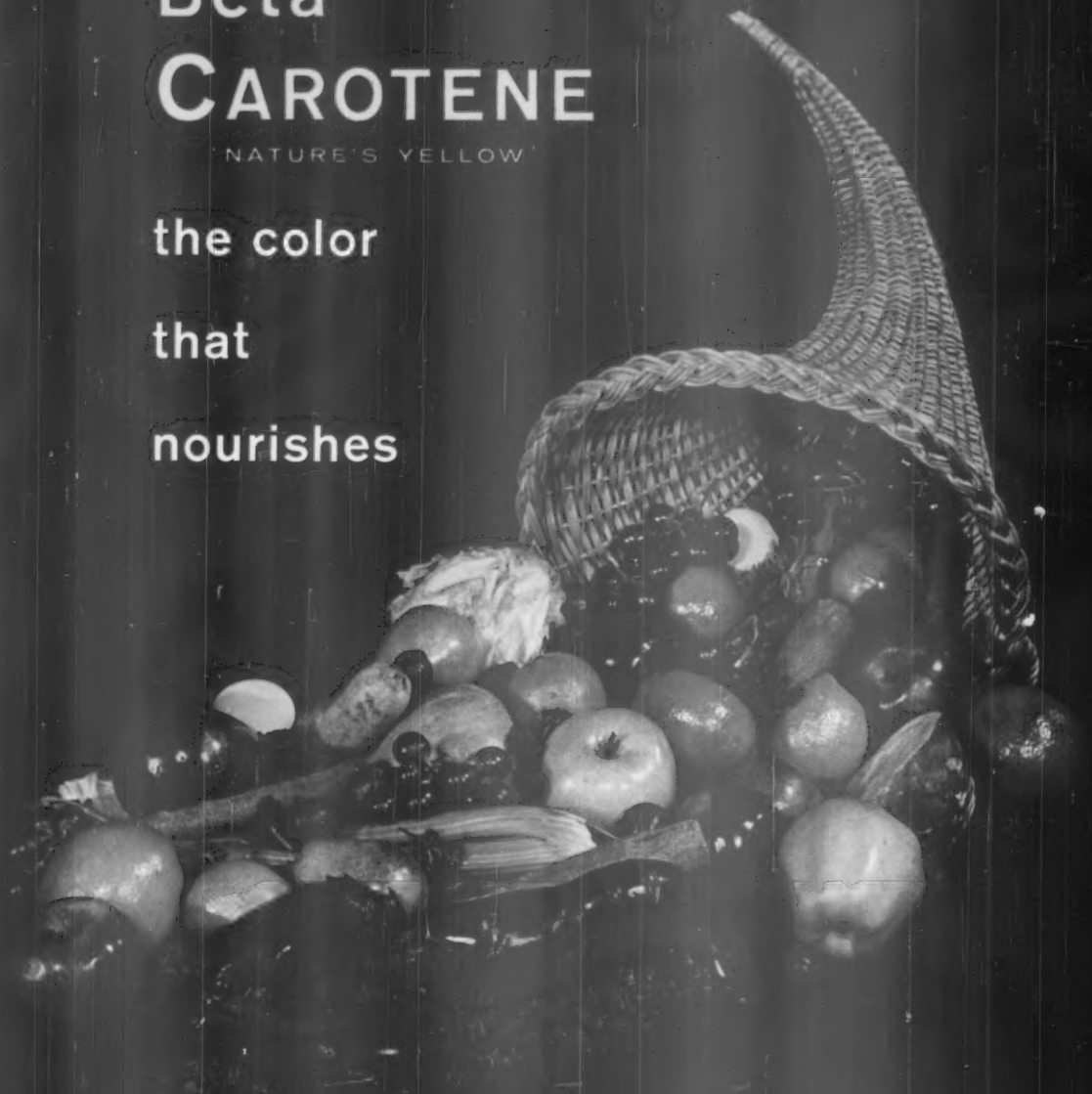

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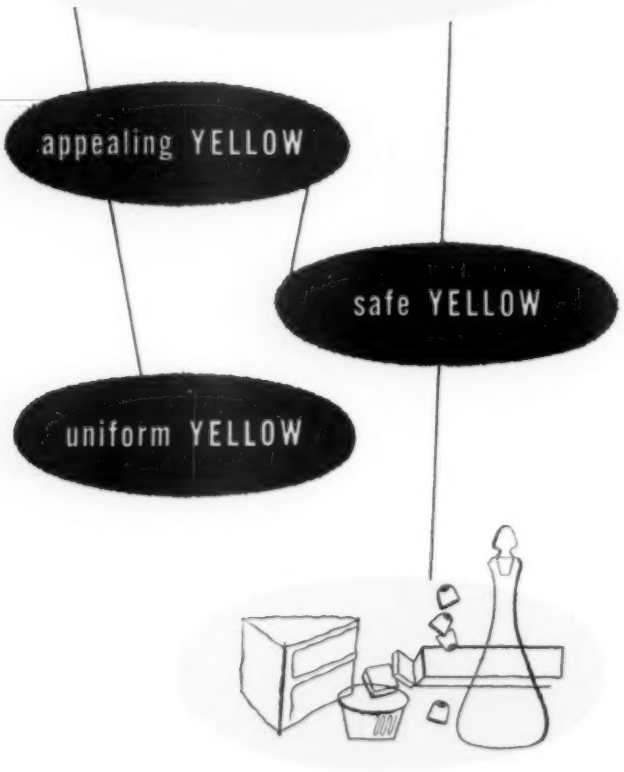

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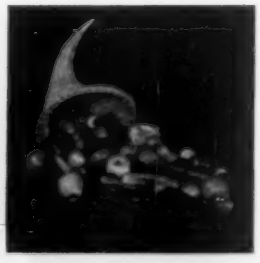

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Use of Calcium Hydroxide for Firming Canned Green and Red Sweet Bell Pepper[a]

(Manuscript received May 9, 1960)

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When red or green sweet bell pepper, *Capsicum frutescens*, is canned by procedures normally recommended for non-acid vegetables, the flesh becomes soft and mushy. This is not necessarily undesirable for some uses such as in sandwich spreads and other similar foods; however, when canned pepper is to be used as a substitute for fresh pepper in salads and various other food items a firmer product is usually more desirable. Since the pH of pepper is above 4.5 it is recommended that it be canned under steam pressure to insure proper sterilization *(3)*.

Use of calcium compounds as firming agents for canned fruits and vegetables is well known *(5, 6, 8)*. Probably the most widely used compounds for this purpose are calcium chloride and calcium sulfate. Calcium hydroxide has also been used as a firming agent; however, its use for this purpose appears to have been generally limited to sliced tomato, cucumber, and melon pickles. The use of minute amounts of calcium hydroxide for controlling color in canned vegetables has also been proposed *(1)*.

Research workers and processors have recognized the need for a firmer canned pepper *(4, 7)*. Powers *et al (7)* found that the firmness of canned pimiento pepper could be improved by acidification with citric acid and the addition of calcium chloride to the contents of the can to equalize at 0.07%. The shorter processing time required, combined with the firming effect of the calcium chloride, resulted in a firmer product. It was reported that the calcium chloride imparted a bitter flavor to the canned pepper when added at the rate of 0.07%.

Because of the bitter flavor that reportedly occurs in canned pepper containing calcium chloride, a different approach was followed in this study from that used by Powers and his group *(7)*. In this investigation, fresh pepper was treated with the firming agents and the residue removed from the surface by rinsing thoroughly with cold water prior to acidification and subsequent heat processing.

EXPERIMENTAL

Green and red sweet bell peppers of the California and Yolo Wonder varieties were used. The pods were cored, washed, cut into halves and diced into ⅜-in cubes in preparation for the firming treatments.

In the initial phase of this study 3 calcium compounds were evaluated and compared as firming agents for canned pepper. These compounds were calcium chloride, calcium sulfate, and calcium hydroxide. In making the comparisons between various firming agents, water was used as a carrier and the equivalent level of calcium was adjusted to 0.3% by weight. The treatments consisted of dipping the pepper cubes in water containing 0.3% calcium for 1 min with agitation. After the 1-min calcium dip, the cubes were removed and allowed to drain for different periods of time up to 60 min followed by rinsing with cold water to remove the calcium residue remaining on the surface of the pepper before heat processing.

The procedure for heat processing the pepper was the same throughout this investigation and consisted of the following steps: (a) Firmed and rinsed pepper cubes were blanched for 2 min. in boiling water, packed hot into No 2 cans and covered with a hot aqueous solution containing 1% citric acid and 2% sodium chloride; (b) the cans were sealed immediately and heated for 10 min in free flowing steam and then cooled with cold water.

After it was determined that calcium hydroxide (lime) was the most effective of the calcium compounds evaluated for firming pepper when utilized as a preheat treatment, it was selected for further evaluation and study. Various methods of applying the lime, duration of treatments and concentrations required for optimum results were evaluated.

Calcium hydroxide was applied by 3 different methods; namely, soaking in lime water, dipping in lime water, and by direct application of the dry material. Soaking treatments consisted of submerging pepper cubes in 0.2, 0.3 and 0.4% lime water with agitation for periods of time ranging from 0 to 60 min followed by a thorough rinse with cold water. The ratio of pepper to lime water was 1:1 by weight. Immediately after the pepper was treated and rinsed with cold water it was heat processed. The second method of lime application consisted of submerging the pepper cubes in 0.2 0.4 and 0.6% lime water for 1 min with agitation. Samples were then removed, allowed to drain for periods of time ranging from 0 to 60 min and rinsed to remove the lime residue. The ratio of pepper to lime water in this case was 1:1½ by weight. The third method of lime application consisted of applying the dry calcium hydroxide directly to the pepper cubes at the rate of 0.1, 0.2 and 0.3% based on the weight of pepper. The lime was mixed with the pepper as evenly as possible and allowed to remain in contact with the cubes for periods of time ranging from 0 to 60 min before rinsing and subsequent heat processing.

When pepper halves were firmed without cubing, samples were covered with lime water in the ratio of 1 lb of pepper to 3 lb of lime water for periods of time ranging from 0 to 3 hr. Concentrations of calcium hydroxide in the lime water used for firming pepper halves were 0.2, 0.3 and 0.4% by weight.

In order to determine whether the calcium hydroxide firming treatments were effective over an extended period of storage, cubed green pepper was firmed by soaking in 0.1, 0.2, 0.4 and 0.6% lime for 10, 20 and 30 min prior to rinsing and heat processing. The canned samples were stored at room temperature, which ranged from 70 to 90° F during the storage period. Firmness evaluations were made after 0, 2, 4 and 6 months.

Firmness was determined with a Christel Texture meter equipped with a drag point indicator and a range of 0 to 160 lb. In making the firmness evaluations, the cell was filled with 82 g of drained canned pepper cubes and the unit operated at a speed of 60 rpm. Each sample was also examined organoleptically to determine whether any off-flavors resulting from the various treatments were evident.

Data were analyzed by the analysis of variance techniques from which linear and curvilinear responses to firming treatments were tested.

[a] Approved by the Director of Research of the North Carolina Agricultural Experiment Station as Paper No. 1160 of the Journal Series.

RESULTS

Comparison of firming agents. Calcium hydroxide was much more effective as a firming agent for canned pepper than either calcium chloride or calcium sulfate (Figure 1). The firmness response of the pepper to calcium hydroxide was very rapid and increased in a curvilinear pattern. Beginning with an average firmness rating of 26 for the control (0 min), a peak rating of 71 was reached after approximately 45 min in contact with the calcium hydroxide. After the peak of firmness was reached, there was a slight but significant decline. The firmness of pepper treated with calcium chloride and calcium sulfide increased very slowly in a linear pattern as the treatment time advanced from 0 to 60 min (Figure 1). Based on the maximum firmness reached with the 3 compounds, calcium hydroxide was more than 6 times as effective as calcium chloride or calcium sulfate for firming pepper.

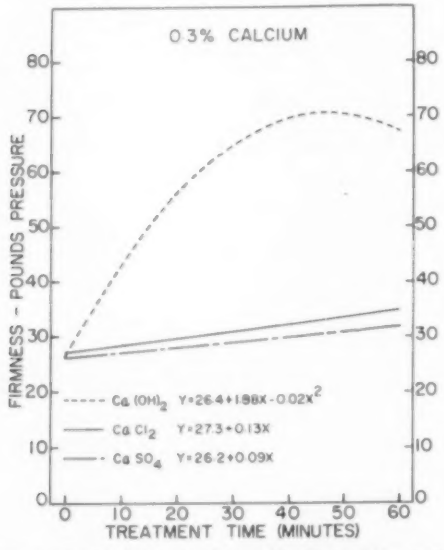

Figure 1. Comparison of calcium chloride, calcium hydroxide and calcium sulfate as firming agents for canned pepper treated at the rate of 0.3% calcium by dipping fresh pepper cubes 1 minute and allowing them to drain for different periods of time. The control or non-treated is indicated by 0 time.

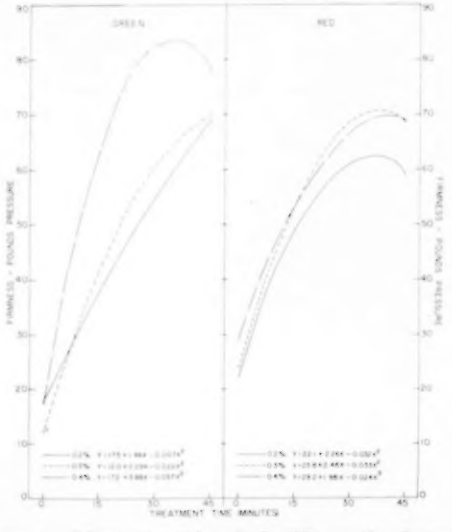

Figure 2. Effect of calcium hydroxide on the firmness of canned pepper when applied to the fresh cubes by soaking for various periods of time at different concentrations. The control or non-treated is indicated by 0 time.

Effect of soaking in lime water. The firmness of green pepper was greatly improved by soaking in lime water (Figure 2). The rate of firming was accelerated as the lime concentration increased. However, the firmness of green pepper firmed with 0.4% calcium hydroxide reached a peak after about 30 min and began to decline.

Calcium hydroxide was also effective in firming cubed red pepper when applied by soaking (Figure 2). Cubes treated for 30 min or longer with 0.3 and 0.4% lime water were firmer than those treated with 0.2%. Relatively little difference was evident in the firmness of red pepper treated with 0.3 and 0.4% lime.

Red pepper halves treated by soaking in lime water became firmer as the treatment duration increased (Figure 3). The response of red pepper halves to lime treatments was not as rapid as that which occurred with the smaller cubes similarly treated (Figure 2). At the end of 3 hr the firmness of pepper halves treated with the 3 different concentrations of lime was about equal; however, the firmness of pepper halves treated with 0.4% lime had reached a peak and was tending to decline.

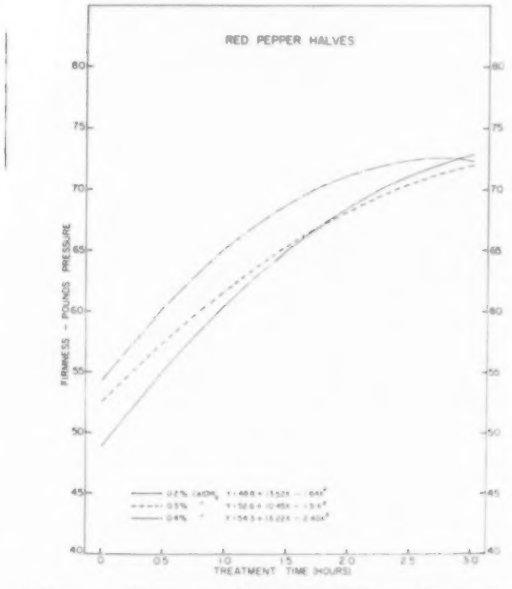

Figure 3. Effect of calcium hydroxide on the firmness of canned pepper when applied to the fresh halves by soaking for various periods of time at different concentrations. The control or non-treated is indicated by 0 time.

Effect of dipping in lime water. When pepper was dipped in lime water for 1 min and allowed to drain, an increase in firmness occurred in green pepper cubes as the lime concentration and duration of treatment advanced (Figure 4). A peak of firmness was reached in cubes treated with 0.6% lime water after about 40 min and after 50 min when treated with 0.4% lime water. Green pepper dipped in 0.2% lime water for 1 min continued to advance in firmness throughout the treatment period.

Red pepper cubes did not respond to the lime treatments as well as the green (Figure 4). However, the firmness did improve considerably in the red pepper as the lime concentration and time increased. A point of maximum firmness was reached with red pepper after approximately 45 min.

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Developed by CARBIDE research just a few years ago, SENTRY Sorbic has become one of the most dependable and widely used mold inhibitors for control of food spoilage. And because research in product improvement never stops at CARBIDE, you can now get a *better* Sorbic—a reliable mold and yeast inhibitor that's pure-white and completely free flowing, with little detectable odor to affect the flavor or aroma of your most delectable foods.

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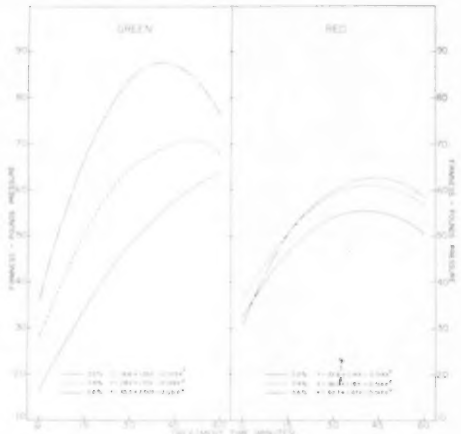

Figure 4. The effect of calcium hydroxide on the firmness of canned pepper when applied by dipping fresh cubes for 1 minute in lime water and allowing them to drain for various periods of time before rinsing. The control or non-treated is indicated by 0 time.

Effect of applying dry calcium hydroxide. When lime was applied directly to the pepper cubes without the use of water as a carrier, there was a very good firmness response (Figure 5). This was particularly true with green pepper. Firmness increased rapidly in green pepper at all levels of lime concentration as the treatment time advanced; however, it was highest in cubes treated with 0.2%.

Red pepper cubes also became firmer as a result of the direct lime treatments, but not to the extent that occurred in green pepper (Figure 5). Samples of red pepper treated with 0.2 and 0.3% lime applied directly to the cubes reached a point of maximum firmness between 40 and 50 min. Red pepper cubes treated with 0.1% lime continued to increase in firmness without reaching an apparent peak during the 60 min in contact with the lime.

Effect of storage. A graphic summary of the effect of lime treatments on the firmness of canned green bell pepper over a period of 6 months is shown in Figure 6. In this phase of the investigation, as in those previously described, the lime-treated samples were much firmer than the control which had not been treated with calcium hydroxide. The regression equation for

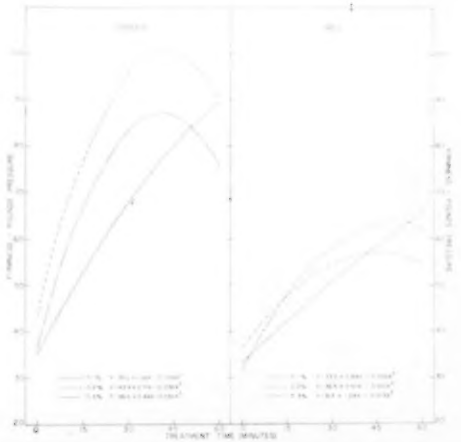

Figure 5. The effect of calcium hydroxide on the firmness of canned pepper when applied dry to the fresh cubes. The control or non-treated is indicated by 0 time.

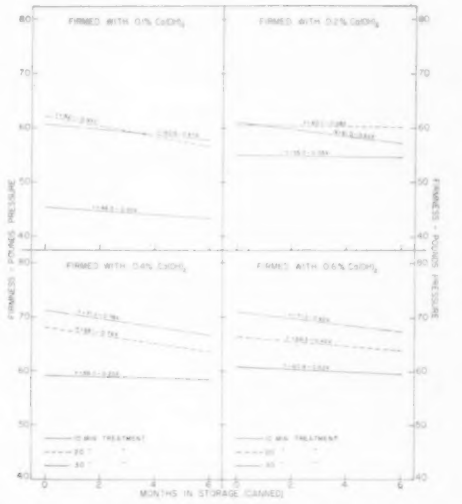

Figure 6. The effect of calcium hydroxide firming treatments and storage on the firmness of canned green pepper.

firmness of the non-treated pepper (control) during storage was $Y = 15.4 - 0.24X$. A comparison of the regression coefficient of the control and those for treated pepper shown in Figure 6 indicates that as the storage time advanced a slight decline in firmness occurred in the treated and non-treated canned pepper at about the same magnitude.

When the canned pepper was examined organoleptically, the presence of off-flavors that could be attributed to lime treatments was not apparent. There was a tendency, however, for cubed pepper to become slightly tough when the firmness rating approached 90 lb or higher.

DISCUSSION

When calcium ions are added to a solution of pectic acid a rigid calcium pectate gel forms *(2)*. The mechanism by which pepper is firmed with calcium hydroxide is probably through the production of calcium pectate by calcium ions and pectic acid or some other pectic substance. The firming of tomatoes by the addition of calcium chloride has been attributed to the formation of calcium pectate *(6)*.

It was observed that if the firmed product was not rinsed thoroughly before blanching to remove the excess lime residue, particularly with the higher concentrations, an adverse softening effect occurred during the blanching process. One probable explanation is that the calcium pectate formed during the firming treatment was dissolved when heated in high concentration of lime water resulting in a softening of the tissue. This hypothesis is based on the fact that calcium and magnesium pectate may be modified when heated with hot alkali.

Throughout the study it appeared that the green or less mature fruits responded more significantly to the firming treatments than red or fully ripe, and yet the response of the control which was not lime treated was more often the reverse. Based on the results obtained by other workers *(2, 5)*, one explanation for this phenomenon of the less mature pepper responding better to calcium treatments might be that the

total pectic substances decrease during maturation of the fruit and at the same time the pectin content increases. The more active response of green pepper to firming treatments as compared to red was probably due to a greater amount of the pectate producing substances in green fruits. However, without more proof one could not rule out the possibility of a greater penetration of calcium ions into green than in red pepper.

When calcium hydroxide is used for firming pepper, it is necessary for it to be in contact with the flesh of the fruit before diffusion and firming action can take place. This is apparently due to the fact that the outer skin of fresh pepper is less permeable to the penetration of the lime. This same factor probably accounted for the more rapid firming which took place with cubed pepper as compared with pepper halves.

SUMMARY AND CONCLUSIONS

A study was made to determine the effect of calcium hydroxide and other calcium compounds on the firmness of canned pepper. Among the compounds evaluated were calcium hydroxide, calcium chloride and calcium sulfate. The effects of various methods of treating, duration of treatments and calcium concentrations on the firmness of canned pepper were evaluated. The influence of storage on the firmness of canned pepper was determined.

Calcium hydroxide was very effective as a firming agent for canned pepper when applied to fresh pepper prior to heat processing. Calcium hydroxide was the most effective firming agent evaluated. Good results were obtained by all three of the methods used to apply the lime treatments. Best results were obtained in cubed pepper when the lime was applied at the rate of 0.2 to 0.4%, by weight, for approximately 30 minutes followed by a thorough rinse to remove the lime residue prior to blanching and heat processing. When pepper halves were treated, it was necessary to extend the treatment time to 2.5 hours or longer for optimum results. Green pepper responded better to firming treatments than red pepper.

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Germicidal Effect of Orange Peel Oil and D-Limonene in Water and Orange Juice.

1. Fungicidal Properties Against Yeast[a]

D. I. Murdock and W. E. Allen

Minute Maid Corporation, Orlando, Florida

(Manuscript received May 16, 1960)

THE INTRODUCTION of frozen orange concentrate in 1945–46 created a new outlet for processed citrus juices and resulted in an unprecedented expansion of this industry. In Florida, for example, during that initial season out of a total orange crop of 49,800,000 boxes, less than 1% was utilized by the frozen concentrate industry. This compares with 1958–59 orange production of 86,000,000 boxes, of which 78% of the fruit was consumed by the concentrators with an additional 2% being processed principally into canned single strength juice and chilled juices *(8)*.

It is estimated that after the extraction of juice from the fruit nearly one-half of the total weight of the fruit remains as refuse consisting of peel, pulp, rag, and seeds. New industries have developed over the years to utilize these enormous quantities of waste material. The essential oil found in the peel of fruit is one of the products recovered from citrus refuse *(1, 3)*. Orange peel oil which is widely used as a flavoring constituent for a variety of food products is referred to as cold-pressed when undistilled, and folded when concentrated by vacuum distillation. Commercial D-limonene, more commonly called stripper oil by the citrus industry, is obtained as a by-product from the manufacture of citrus molasses *(1, 3)*. It is used as a penetrating oil, base for soap perfumes, and as an ingredient for clear plastics. Some of the chemical and physical properties of these two oils are shown in Table 1.

[a] Presented at the Twentieth Annual Meeting of the Institute of Food Technologists, May 16, 1960, San Francisco, California.

TABLE 1

Chemical and physical properties of cold pressed orange oil and stripper oil (commercial D-limonene)[1]

	Stripper oil	Florida C.P. orange oil
D-Limonene content—%	95–98	90–95[2]
Aldehyde content—%	0.47–1.50	0.92–2.04
Ester content—%	0.07–2.46	0.04–1.63
Evaporation residue—%	0.03–0.79	1.07–4.93
Specific gravity 25° C/25°C	0.840–0.843	0.842–0.846
Refractive index n_D^{20}	1.4713–1.4721	1.4718–1.4743
Optical rotation a_D^{25}	+95.55–98.90	+95.16–98.05

[1] See *Essential Oils from Florida Citrus* by J. W. Kesterson and R. Hendrickson, *Bull. 521*, Univ. of Fla. Agr. Expt. Sta., Gainesville, Fla. (1953).

[2] D-Limonene content of C.P. orange oil by personal communication from J. W. Kesterson.

Many of the essential oils possess antimicrobial properties *(5, 6)*. Maruzzella and Henry *(5)* investigated the germicidal activities of 100 perfume oils against a series of pathogenic and nonpathogenic bacteria and fungi. They noted that every oil tested possessed remarkable lethal properties with greater activity against fungi than bacteria. Their investigation did not include orange oil or commercial D-limonene. Lord and Husa *(4)* noted that a 1 to 500 dilution of orange oil did not completely inhibit mold growth when this dilution of oil in agar plates was inoculated with syrup containing a mixed culture of molds. The disinfectant power of lemon and orange essences in aqueous solutions against spore-bearing organisms was tested by Piacentini *(7)* and found to be greater than that of phenol. Hahn and Appleman *(2)* found that cold-pressed orange oil exerted a toxic effect upon *Streptococcus faecalis*. This organism was destroyed by concentrations of peel oil of 1,000 ppm (0.1%) and was adversely affected in a decreasing manner by 100 ppm (0.01%) and 10 ppm (0.001%). Peel oil present in concentrations of 1 ppm had no apparent effect against *Streptococcus faecalis*. Little or no specific information on the survival of yeast in orange peel oil and commercial D-limonene was found in the literature.

The purpose of this paper was to investigate the antimicrobial activity of orange peel oil and commercial D-limonene in water and in orange juice at various pH levels against (a) fungi and (b) lactic acid bacteria. Part 1 will be presented at this time.

PROCEDURE

Orange juice used in this study was prepared from 42° Brix Valencia orange concentrate packed by Minute Maid Corporation, Plymouth, Florida. It was reconstituted with tap water to single strength juice by the addition of 3 volumes of water to 1 of concentrate, producing a juice having a Brix value of 12.14° and Brix/acid ratio 15.18:1. After placing 100 ml aliquots of the reconstituted orange juice into 250 ml Erlenmeyer flasks, the flasks were cotton-stoppered, over wrapped with paper and sterilized in flowing steam for 10 min. The juice after sterilization had a pH value of 3.70 and only a trace of oil was present as determined by standard clevenger distillation. Distilled water (pH 7.0) was handled in Erlenmeyer flasks in 100 ml portions in the same manner as was the juice except that it was sterilized in the autoclave for 15 min at 15 lb pressure (250° F).

Orange peel oil (cold-pressed) and commercial D-limonene (stripper oil) were obtained during the 1959 Valencia season and were of commercial grade. They will be referred to in the remainder of this paper as orange oil and stripper oil.

The yeast used in this study, *Zygosaccharomyces major*, was obtained from the culture collection of Continental Can Com-

pany, Chicago, Illinois. It originally was isolated from orange juice concentrate. The test organism was grown on orange serum agar slants prepared in 8-oz screw-cap bottles and incubated 48 to 72 hr at 30° C (86° F). Each slant was washed with 10 ml of sterile water and the suspensions were then combined and placed in a sterile bottle containing glass beads. The number of organisms present in this suspension was determined by making plate counts on orange serum agar after 72 hr of incubation at 30° C (86° F). The suspension was stored in a refrigerator when not in use.

The survival of the test organism in distilled water and in orange juice was determined by inoculating the test medium in the Erlenmeyer flask so that each ml after inoculation contained approximately 1,000,000 yeast cells per ml. The actual level of inoculum was determined by plating the solution after it had been allowed to agitate 3 to 4 min on a magnetic stirrer. Then by means of a sterile micropipette, the desired level of each oil was added (0.02 and 0.05 and 0.1 ml, respectively, equivalent to 0.02%, 0.05% and 0.1% oil by volume). A control which was inoculated with yeast but contained no oil was also included with each test run. The flasks remained on magnetic stirrers under constant agitation throughout the test period. Plate counts were made in duplicate at hourly intervals for the first six hours. Some of the orange juice variables were also plated after a 24-hr period. Orange serum agar was used as the plating medium and all plates were counted after 72 to 96 hr of incubation at 30° C (86° F). Each test was made at room temperature which was approximately 25° C (77° F).

RESULTS AND DISCUSSION

The survival of yeast *(Zygosaccharomyces sp)* in distilled water (pH 7.0) at oil levels of 0.02%, 0.5%, and 0.1% is shown in Figure 1 for orange oil, and in Figure 2 for stripper oil. Both oils, as expected, exhibited their greatest anti-microbial properties at the 0.1% level.

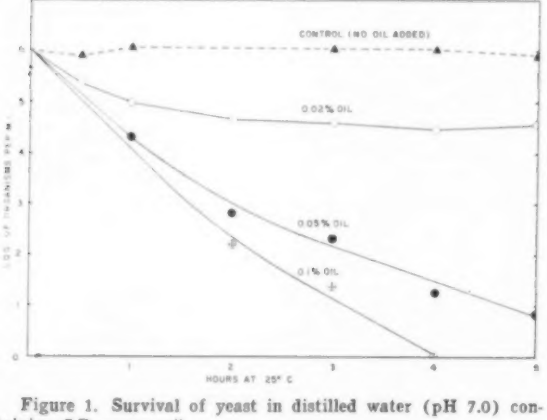

Figure 1. Survival of yeast in distilled water (pH 7.0) containing C.P. orange oil.

Orange oil is customarily added to orange concentrate, both frozen and hot-packed, in the range of 0.01% to 0.02% by volume (reconstituted basis), to enhance the flavor of the product. A study was made to determine if a concentration of 0.02% orange oil in juice was toxic to the test organism. A comparison was also made with stripper oil. From the data (not shown in this paper) it was apparent that 0.02% orange or stripper oil had no adverse effect on yeast during the first 6 hr at 25° C (77° F) but growth

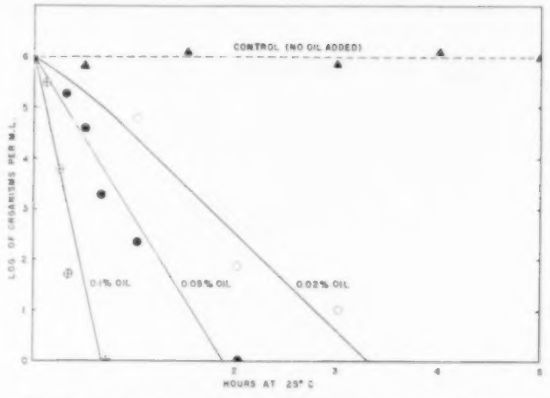

Figure 2. Survival of yeast in distilled water (pH 7.0) containing stripper oil.

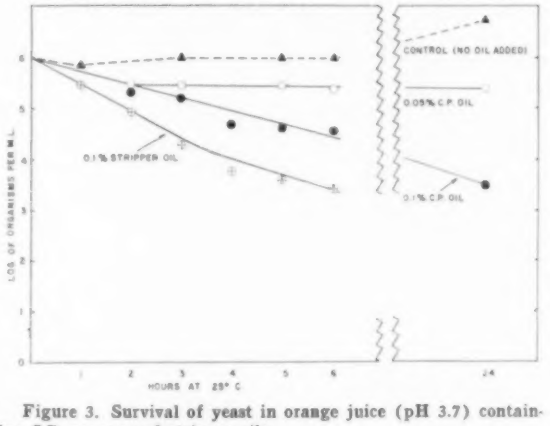

Figure 3. Survival of yeast in orange juice (pH 3.7) containing C.P. orange and stripper oils.

occurred thereafter. On the other hand, results in Figure 3 indicate no yeast growth or change in microbial population occurred in the product containing 0.05% orange oil after being held 24 hr at 25° C (77° F). Stripper oil was not tested at this oil level. Both oils at a concentration of 0.1% in orange juice were lethal to yeast which died at a steady rate, as the data in Figure 3 indicate. Stripper oil in water as well as in orange juice was more toxic to the test organism than orange oil at any given concentration.

A study was made to determine if a relationship exists between the pH of orange juice and the antimicrobial properties of orange and stripper oils. The orange juice was obtained from the same lot of orange concentrate used for the previous experiments. It was reconstituted with tap water and adjusted with anhydrous citric acid for values below pH 3.70, and with 4N NaOH for values above pH 3.70. The juice after the desired pH values were obtained was handled in Erlenmeyer flasks as previously described. Sterilization was in flowing steam for product below pH 5.0, and for 15 min in the autoclave at 15 lb pressure (250° F) for juice above this pH value. The survival of the test organism in orange juice adjusted to pH

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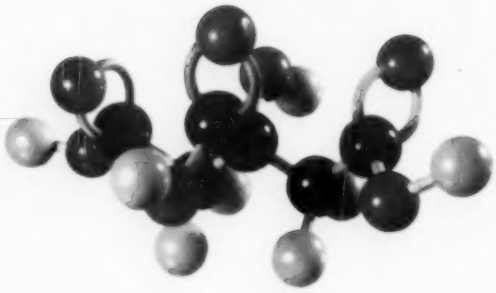

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Indeed, Pfizer Citric Acid has been a key product to the food industry for many years. The words "citric acid" on countless labels have established it as a familiar and trusted ingredient.

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How Pfizer Pioneered in Production of Citric Acid in America

While the advantages of citric acid as a food ingredient have long been appreciated, the supply was at one time quite limited. Up until 1880, virtually all the citric acid used in this country was made in Europe. In that year, Pfizer conceived the idea of importing the raw materials from abroad and producing citric acid in the United States. The results were successful and Pfizer gradually became a leading manufacturer of this important commodity.

But production still depended on raw materials supplied from overseas. In a far-sighted decision around the turn of the century, Pfizer began to experiment with methods of producing citric acid from domestic materials. In 1914, this research program focused on the possibility of fermenting citric acid from a sucrose base. Five years later, Pfizer opened a pilot plant employing such a process.

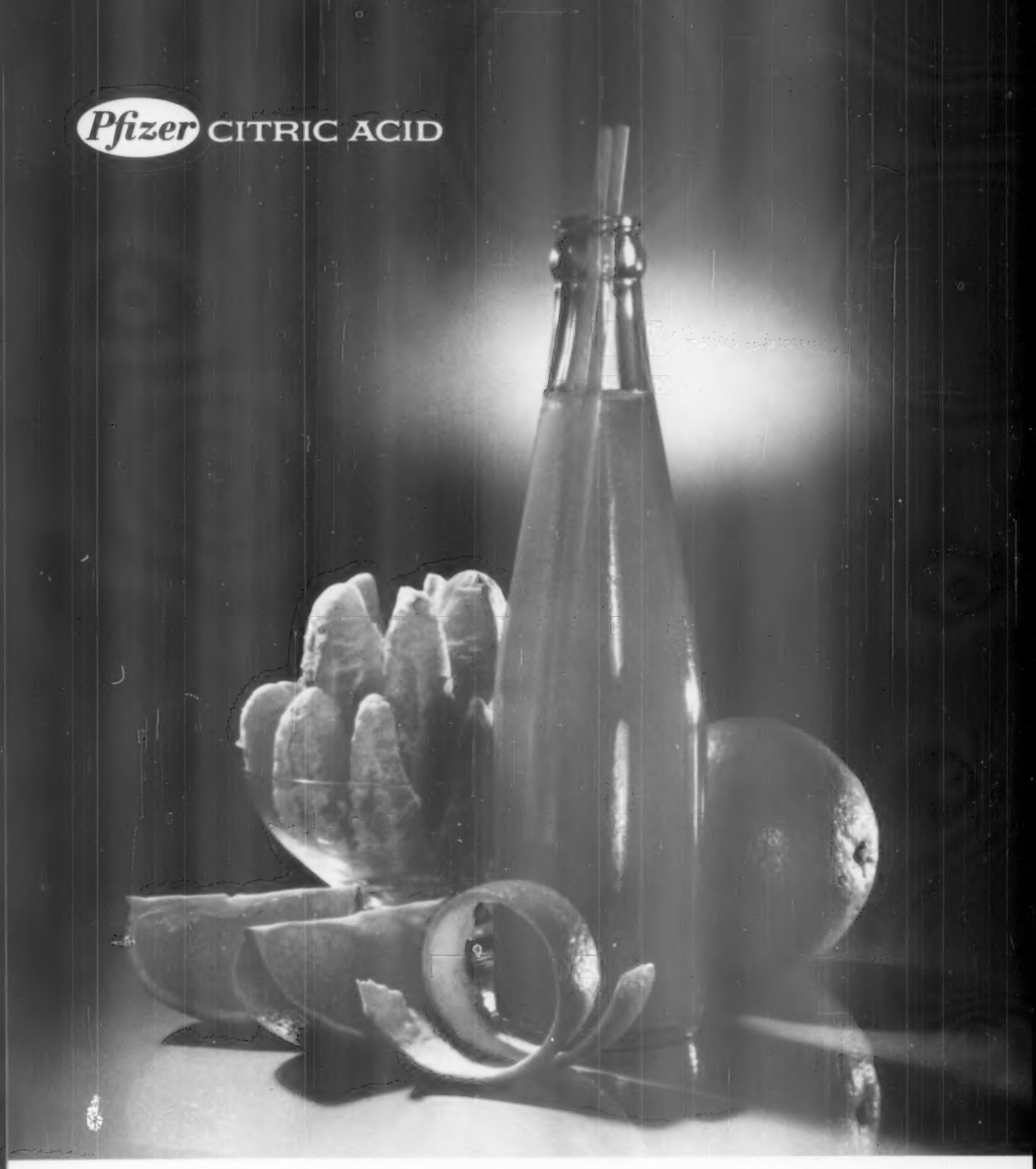
By 1923 the company was convinced that it had really found the formula for producing citric acid of the highest quality on an economical basis. A commercial production plant was put into operation and in 1924 American industry was receiving regular deliveries.

The Pfizer fermentation process proved so efficient that by 1939 the price of citric acid had been reduced to ¼ the pre-World War I price!

The American food industry was thus assured a readily available supply of economical citric acid from a dependable domestic source.

In addition, by enlisting the assistance of the Pfizer Technical Service staff, customers have shared in Pfizer's extensive knowledge of citric acid and effected sizeable time and money savings in product development and improvement.

You will see on the pages that follow some of the products in which this versatile acidulant serves as a key ingredient—ample evidence of the importance of citric acid in today's food industry!



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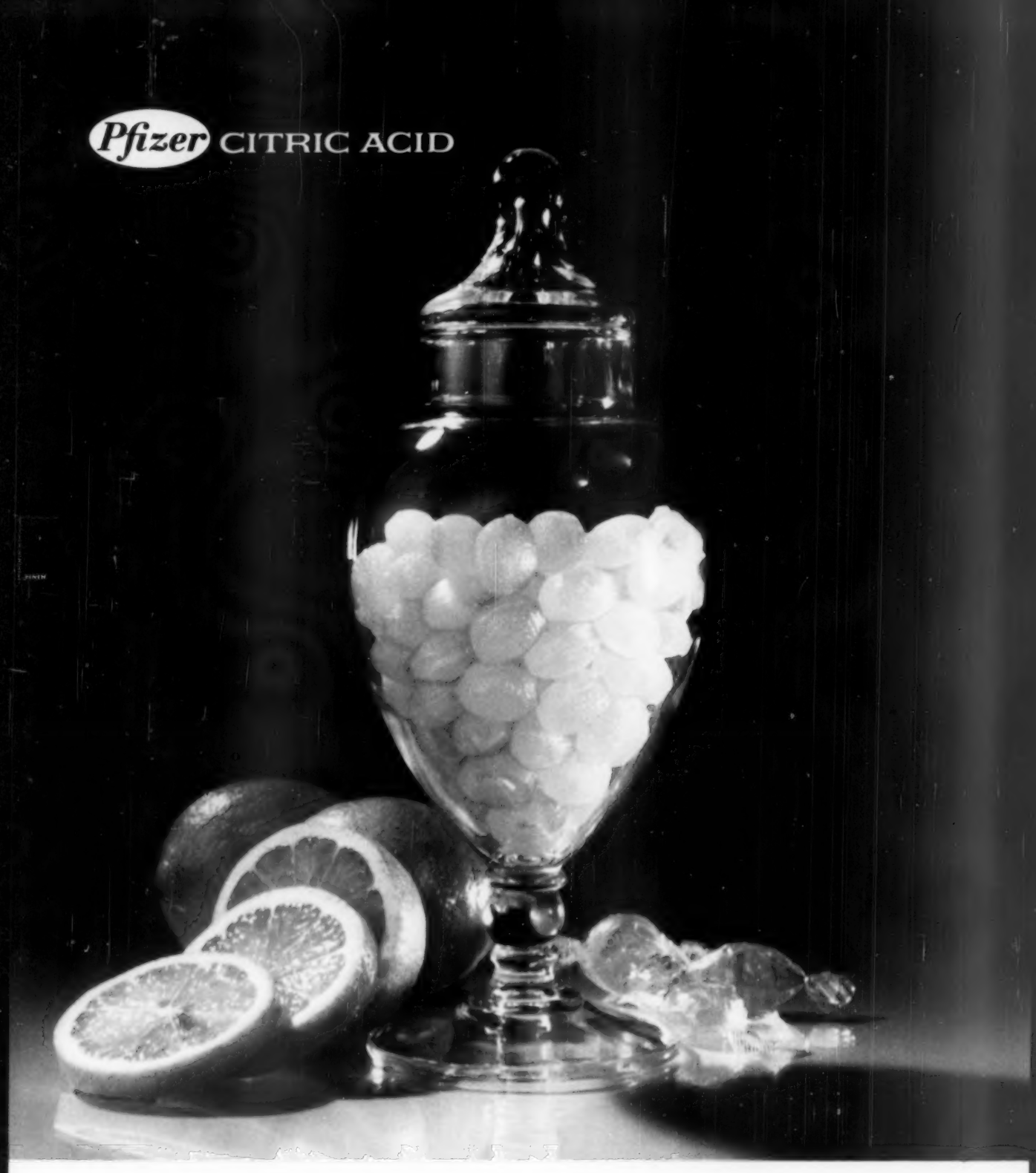

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Citric acid gives fruit-flavored desserts that natural tartness!

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Citric acid's flavor-benefit is that "little extra" so important to true fruit taste!

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Citric acid gives a natural "lift" to real-fruit flavors!

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Citric acid helps bring out the tart-sweet goodness of fresh fruit!

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TRADITIONALLY BEST FOR BEVERAGE POWDERS

Citric acid adds the tanginess that contributes to full, rich flavor!

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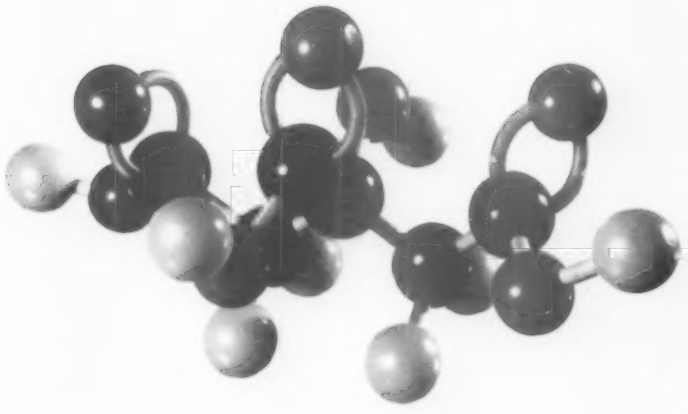
WHERE YOU MAY BE USING CITRIC ACID TOMORROW

In the future, uses for citric acid will extend beyond the area of fruit-flavored products. One area where citric acid may soon prove important is vegetables, particularly canned vegetables.

Small amounts of citric acid help control the pH level in canned tomatoes, beans, carrots and asparagus. Experiments in canned, whole tomatoes indicate that by controlling the pH with citric acid it may be possible to reduce processing time and/or temperature, resulting in a firmer whole tomato.

It has also been shown that citric acid sequesters trace metal contaminants in the pack, thus contributing to the quality of canned vegetables.

Pfizer, world's leading producer of citric acid, will continue to keep you informed on valuable new developments concerning citric acid—nature's acidulant.



Pfizer

CITRIC ACID

TECHNICAL INFORMATION

Pfizer offers citric acid in two basic forms—anhydrous and hydrous

CITRIC ACID, U.S.P., ANHYDROUS

This is produced as colorless, translucent crystals or as a pure white powder. It is odorless and has an acid taste. The compound is stable in the presence of light and air and resists caking under normal storage conditions. Because there is no water of crystallization present, anhydrous citric acid is particularly economical. Its reduced bulk leads to lower costs for handling, shipping, and storage. Furthermore, the lack of water of crystallization assures you of a uniform product and permits exact quality control.

CITRIC ACID, U.S.P., HYDROUS

This form of citric acid has been available for many, many years and is still counted on as a high quality acidulant for a wide variety of products. In physical appearance, taste and odor, it is similar to the anhydrous compound. However, it contains 1 mol water of crystallization (8.58% moisture). Stable in the presence of light and air, it will tend to lose water at high temperatures or in a dry atmosphere.

Various salts and esters of citric acid also find widespread application in the food industry. Two of the more prominent, sodium citrate and Citroflex® A-4, are described below.

SODIUM CITRATE

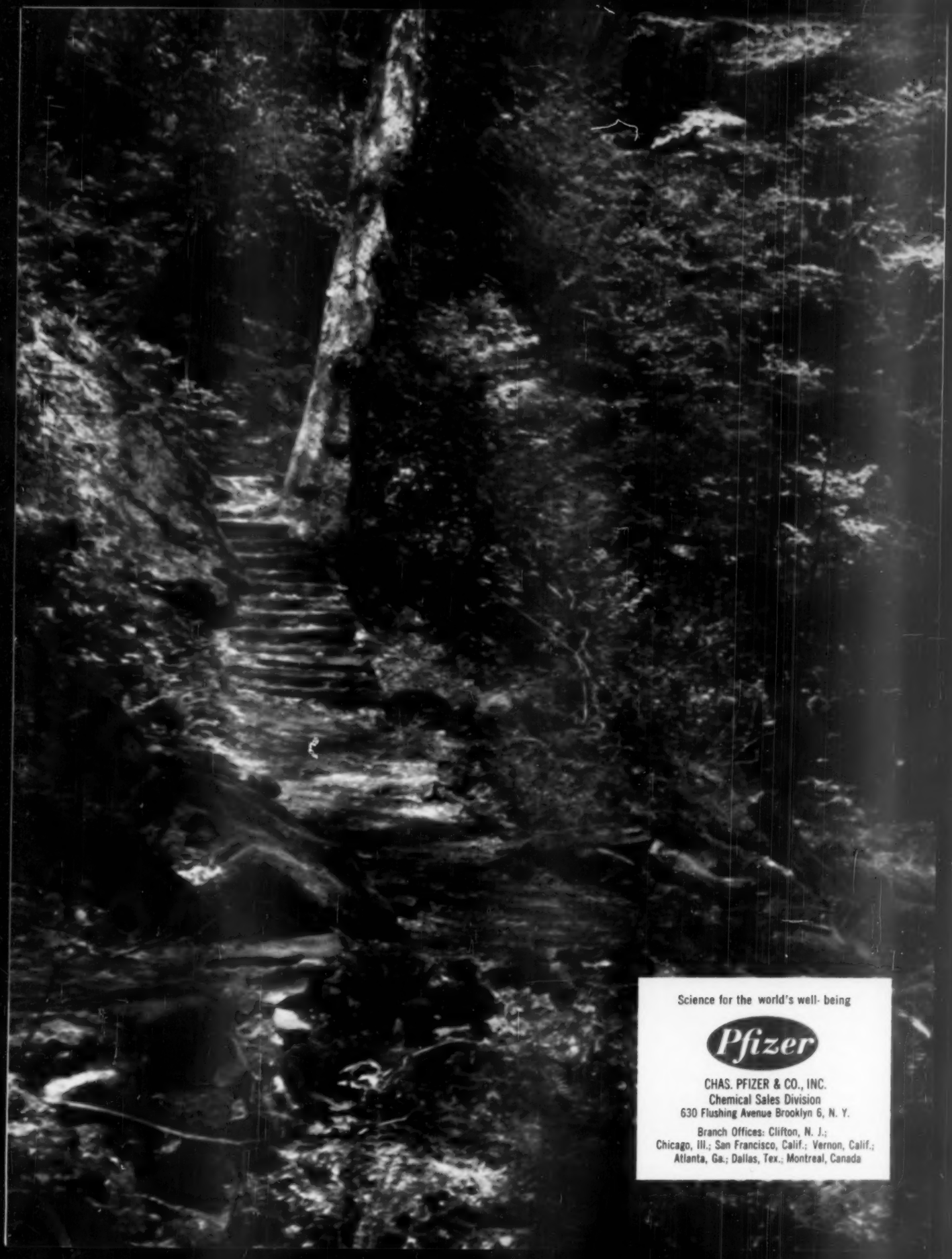
This sodium salt of citric acid is a versatile, easily assimilated product that has many uses in the food industry. Used as an emulsifier in cheese products, it produces smooth, even melting cheese that slices and spreads easily. Similarly, it improves the texture of ice cream. Pfizer Sodium Citrate acts as a buffer salt to control the pH in jellies, preserves, dessert powders, and canned fruit juice blends. And like citric acid, it also brings out true fruit flavor.

CITROFLEX® A-4 PLASTICIZER

Non-toxic, odorless, Pfizer Citroflex A-4 has been accepted by the Food & Drug Administration for use in packaging for both fatty and non-fatty foods. Citroflex A-4 (Acetyl Tributyl Citrate) shows excellent results in plasticized vinyl films for meat wrapping, vinyl coating for food containers and hot drink cups, and vinyl plasticizers for food jar liners and sealing rings. In addition, Pfizer Citroflex A-4 is excellent in polyvinyl acetate adhesives. Low in cost and high in performance Pfizer Citroflex A-4 is finding wide use in plastic coatings for the food industry.

PHYSICAL PROPERTIES OF PFIZER CITRIC ACID

	Anhydrous	Hydrous
Formula	$C_6H_8O_7$	$C_6H_8O_7 \cdot H_2O$
Molecular Wt.	192.12	210.14
Density	1.67	1.54
Melting Point	153°C	Loses water
Solubility, 25°C		
Water	161.8 gm./100 ml.	208.6 gm./100 ml.
Alcohol	59.1 gm./100 ml.	90.4 gm./100 ml.
Ether	0.75 gm./100 ml.	1.6 gm./100 ml.



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3.0, 3.50, 4.0, 5.0, 6.0 and 7.0 was studied. However, for sake of simplicity, only the results obtained at pH 3.0, 4.0, 6.0, and 7.0 are reported in this paper (chemical analyses of these juice variables are shown in Table 2).

The survival of yeast in orange juice containing 0.1% orange oil at the above pH values is shown in Figure 4, and for 0.1% stripper oil in Figure 5. From the data it will be noted that in the control juice samples (product inoculated with the test organism only) little or no yeast growth occurred at 25° C (77° F) in 6 hr, but after 24 hr a sizeable increase in total viable count was obtained. A concentration of 0.1% orange oil in juice showed no appreciable difference in toxicity on yeast between pH 3.0 and 4.0, and between pH 6.0 and 7.0. However, the lethal action of the oil on this organism was greater at the two higher pH values than it was at the lower levels. On the other hand, there was a much sharper stepwise correlation between pH of the juice and the toxic effect of 0.1% stripper oil on yeast; the oil being more lethal as the pH of the juice increased. For example, in one test, orange juice adjusted to pH 3.0 with citric acid and containing 0.1% stripper oil had a yeast population of 100,000 organisms per ml 3 hr after inoculation with 1,000,000 yeast cells per ml. Another sample of juice adjusted to pH 7.0 with NaOH contained no viable organisms on a 1 to 1 dilution plate after a like period. Similar results were also obtained 24 hr later indicating the juice was apparently sterile.

A comparison was made of the two oils to show the time in hours at 25° C (77° F) required to obtain a 99.9% reduction in the yeast population (1,000,000 yeasts per ml to 1,000 organisms per ml). These relationships for water (pH 7.0) and for reconstituted orange juice (pH 3.70) at oil levels of 0.02, 0.05, and 0.1% are shown in Table 3. To obtain a 99.9% reduction in yeast population with 0.1% orange oil, it required 1.6 hr in water in comparison with 33 hr in orange juice; for stripper oil, 0.4 hr in water in contrast to 7.2 hr in orange juice. A similar comparison of lethality values for yeast in orange juice adjusted to various pH levels is shown in Table 4 for 0.1%

TABLE 2

Chemical analysis of reconstituted orange juice adjusted to various pH levels by addition of sodium hydroxide and citric acid

pH	° Brix	% Citric acid[1]
3.0	12.17	2.0
3.7[2]	12.14	0.80
4.0	12.11	0.96
6.0	11.91	0.16
7.0	11.89	0.04

[1] Anhydrous citric acid by weight.
[2] Control—reconstituted orange juice with no pH adjustment. Juice adjusted to pH 3.0 with crystalline anhydrous citric acid, and with 4N NaOH for values above pH 3.7.

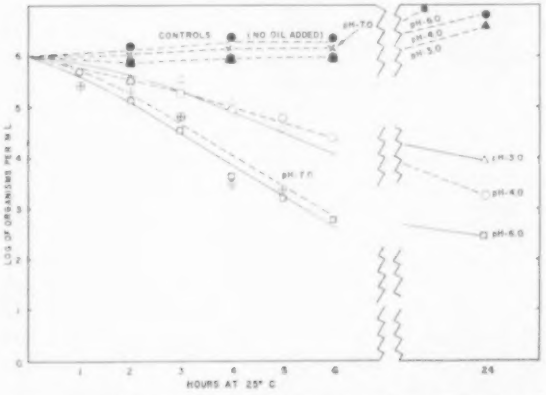

Figure 4. Survival of yeast adjusted to various pH levels and containing 0.1% C.P. orange oil.

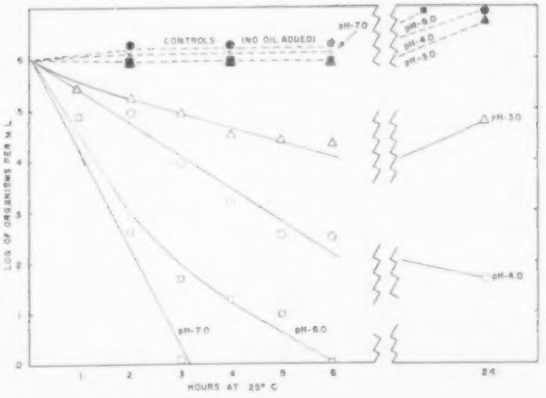

Figure 5. Survival of yeast in orange juice adjusted to various pH levels and containing 0.1% stripper oil.

TABLE 3

Hours required to obtain 99.9% reduction of initial inoculum of 1,000,000 yeast/ml in distilled water (pH 7.0) and in orange juice (pH 3.7)

Comparison of lethality between C.P. orange oil and stripper oil				
Concentration of oil tested	Hours at 25°C			
	Orange oil		Stripper oil	
Per cent	Water	Orange juice	Water	Orange juice
0.02	>5	....[1]	1.8	....[1]
0.05	2.0	....[2]	0.9	....
0.10	1.6	33[3]	0.4	7.2[3]

[1] 0.02% oil had no adverse effect against yeast.
[2] Reduction of 99.9% not obtained in 24 hours; yeast population showed no change from 6-hour period.
[3] By extrapolation of curve on graph in Figure 3.

TABLE 4

Hours required to obtain 99.9% reduction of initial inoculum of 1,000,000 yeast/ml in orange juice adjusted to various pH levels

Comparison of lethality between C.P. orange oil and stripper oil				
	Hours at 25°C			
	pH 3.0	pH 4.0	pH 6.0	pH 7.0
0.1% C.P. Oil	>24	>24	5.4	5.8
0.1% Stripper Oil	(6)*	4.7	2.0	1.6

* Reduction of 99.9% not obtained in 6 hours; growth occurring at 24 hours.

concentration of each oil. The data indicate that for orange oil more than 24 hr were required to obtain a lethality of 99.9% at pH 4.0 in contrast to 5.8 hr at pH 7.0. On the other hand, stripper oil required 4.7 hr at pH 4.0 in comparison with 1.6 hr at pH 7.0.

It was noted during the course of this investigation that orange oil enhanced the fungicidal effect of sodium benzoate against the test organism. This is shown in Figure 6 for distilled water, and in Figure 7 for reconstituted orange juice (pH 3.70). The data show that 0.01% sodium benzoate in water had no apparent effect on yeast while orange oil in a concentration of 0.02% was only slightly toxic to this organism. On the other hand, by adding both compounds to water at the above concentrations a decided increase in lethal action against the test organism was

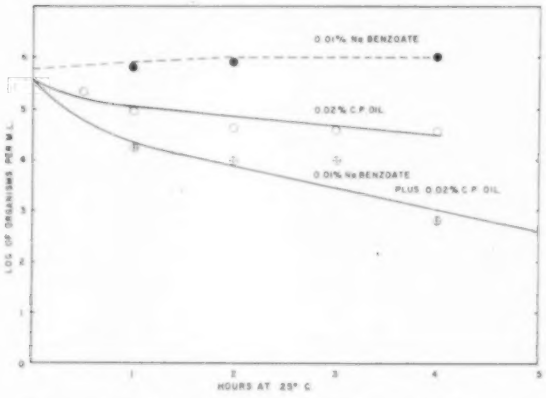

Figure 6. Survival of yeast in distilled water (pH 7.0) containing sodium benzoate and C.P. orange oil.

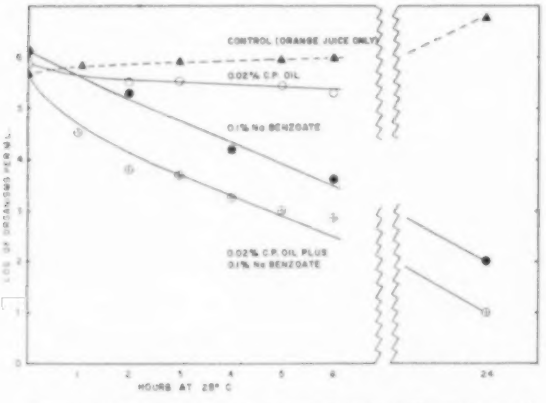

Figure 7. Survival of yeast in orange juice (pH 3.7) containing sodium benzoate and C.P. orange oil.

obtained. Stripper oil in water in combination with benzoate was even more effective against yeast than orange oil. A similar relationship between benzoate and oil is shown in Figure 7 where these materials were added to orange juice (pH 3.70). In this test, orange juice containing 0.02% orange oil and 0.1% sodium benzoate exhibited a greater lethality against the test organism than the combined individual toxic effects of each compound in orange juice.

The synergistic effect of orange oil on sodium benzoate is of extreme interest because it might make possible use of lower levels of benzoate and thus minimize off-flavors typical of that compound.

During the study there was no evidence that the concentration of orange or stripper oils introduced into the plates while making serial dilutions were fungistatic to the test organism.

As matter of interest, the survival of the test organism in orange juice containing 0.1% refined peanut oil was determined and found to exhibit no lethal properties (Table 5).

TABLE 5

Survival of yeast in orange juice pH 3.7 containing 0.1% refined peanut oil

Number of hours at 25° C	Microorganisms per ml
0	835,000
1	775,000
2	1,040,000
3	1,190,000
4	1,200,000
5	1,090,000
6	1,320,000

As previously stated, stripper oil was more toxic against *Zygosaccharomyces sp* under all conditions investigated than orange oil. The hydrogen ion concentration of the test medium had a greater influence on the lethality of stripper oil than it did on orange oil. The mechanism of action of the antimicrobial activity of the two oils studied is not known nor is the apparent synergism with sodium benzoate. It is suspected D-limonene is the principal antimicrobial component since this compound is present in such high concentrations in both orange oil (90 to 95%) and stripper oil (95 to 98%). However, it is not clear why stripper oil should be so much more lethal than orange oil.

Maruzzella and Henry *(5)* in their studies on the antimicrobial activity of perfume oils suggest that common toilet articles such as soaps, creams, shampoos, lotions, ointments, etc., be treated with perfume oils which when applied externally for various hygienic purposes would kill microbes in the area of application. It is entirely possible that orange oil and stripper oil may have similar application. Also the use of small quantities of stripper oil in paints to prevent mildew may have potentialities.

It is hoped that the data presented in this paper will arouse interest in the commercial application of these oils as antimicrobial agents.

SUMMARY

Fungicidal effect of orange peel oil and D-limonene (stripper oil) was determined against *Zygosaccharomyces major* in water and in single strength orange juice at 25° C. Both oils exhibited lethal properties in water (pH 7.0) in concentrations as low as 0.02%,

while in orange juice 0.1% was required. D-limonene was more effective than orange peel oil in both water and orange juice.

The fungicidal properties of these oils in orange juice adjusted to various pH levels were determined. It was found that orange peel oil and D-limonene were more effective at pH 6.0 and 7.0 than they were at pH 3.0 and 4.0. Orange juice adjusted to pH 7.0 with NaOH and containing 0.1% D-limonene was sterile 3 hours after being inoculated with 1,000,000 yeast cells per ml. In juice adjusted to pH 3.0 with citric acid, there were 100,000 viable cells per ml remaining at the end of a like period.

Results of this study indicate that the preservative properties of sodium benzoate can be greatly enhanced by the addition of orange peel oil and D-limonene in concentrations as low as 0.02%. It appears that these oils are synergistic to sodium benzoate.

Acknowledgment

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Effects of Partial Pumping on the Shrinkage, Rancidity Development, and Palatability of Aged Hams*

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(Manuscript received April 25, 1960)

AGED HAMS are often not uniform in quality. They sometimes shrink as much as 30% during the aging period *(2)*. New techniques are needed to make a more uniform product and to reduce shrinkage. Pumping is used in curing hams to hasten the introduction of curing ingredients into all the tissues. This effectively reduces spoilage, curing time, and shrinkage and enables a packer to produce a more uniform product. However, pumping is generally considered as unsuitable for hams to be aged. Hams for aging are usually dry-cured. It has been shown *(4, 5)* that the concentration of salt in the outside inch of dry-cured hams may be as much as 10 times greater just after curing than the concentration in the center of the hams. Partial pumping should partially equalize this distribution. This project was devised to test whether a combination of pumping and dry-cure would enable one to produce satisfactorily a more uniform product with less shrinkage.

EXPERIMENTAL METHODS

Sixty-four regular style hams from Hampshire and Duroc hogs were divided into 8 groups. Groups 1d, 2d, 3d, and 4d were dry-cured; groups 1p, 2p, 3p, and 4p (which were paired mates of hams in groups 1d, 2d, 3d, and 4d respectively) were cured by a combination of pumping and dry-curing. The dry-cured hams were cured with a mixture of 7½ lb salt, 2½ lb white sugar, 2 oz potassium nitrate, and 1 oz sodium nitrite per 100 lb hams. This mixture was applied in 3 applications at 3-day intervals. A pumping solution was prepared by mixing the above dry mixture with water to form an 85° pickle. Hams in groups 1p, 2p, 3p, and 4p were pumped to 4, 6, 8, and 10% respectively of their weights and then rubbed with enough of the dry mixture to bring the total cure to the amount used on the dry-cured hams.

All hams were cured for 21 days at 36–40° F (2.2–4.4° C). They were then smoked at a smokehouse temperature of 100° F (37.8° C). They were aged at 65° F (18.3° C) with a relative humidity of 55–60% for 6 months. Hams were weighed to the nearest 0.01 lb while fresh, after pumping, after curing, after smoking, and after each month of aging. Fat samples were removed from the skin side of the fresh and smoked hams and after 2, 4, and 6 months' aging. After 6 months' aging, the hams were cut, photographed, and observed for soundness and aroma. Two center slices were used for palatability studies and a third was used for salt (NaCl) analysis. Seam fat was removed from the shank portion for analysis.

All fat samples were vacuum rendered as described by Kemp *et al (2)*. Characteristics of external fat were determined by iodine-numbers, peroxide numbers, and free fatty acid values. Free fatty acid values and peroxide numbers were determined on the 6-month seam fat. Salt analysis was determined as outlined by Blumer *(1)*. Ham slices for the palatability study were broiled in an electric oven. The *semimembranosus, semitendinosus, and biceps femoris* muscles were used for tasting by 7–10 panel members, with each panel member receiving duplicate samples of the same muscle at any one test. They were scored on a hedonic scale for flavor, saltiness, tenderness, and over-all satisfaction.

Analysis of variance and t-distribution as described by Snedecor *(3)* were used for statistical analysis.

RESULTS AND DISCUSSION

Shrinkage. Table 1 shows the average shrinkage values for all groups and periods. Shrinkage significantly increased each month in both dry-cured and pumped hams. Pumping significantly reduced shrinkage during curing, smoking, and at each period of aging. Although there were considerable differences among groups for shrinkage of dry-cured hams the differences were not significant. This variation was probably due to the fatness of the hams, as the hams in groups 1p and 1d were fatter than those in other groups. There were no significant differences among pumping rates for the pumped hams.

Iodine numbers. Iodine numbers were not significantly affected by time, treatment, or interaction. Table 2 gives the mean iodine values for all groups.

Peroxide numbers. Peroxide numbers for external fat increased significantly with aging in both pumped and dry-cured hams. They were rather erratic within groups as aging proceeded, with little difference due to curing procedure or pumping rate. Table 3 gives a summary of peroxide numbers.

The mean peroxide numbers for seam fat of hams aged 6 months was much lower and less erratic than for external fat. The level of pumping did not significantly affect peroxide values of seam fat. However, as the pumping level increased the peroxide number tended to increase. Table 4 shows these values. Fat from dry-cured hams showed significantly higher peroxide numbers in all groups except group 4.

Free fatty acids. Free fatty acid values of external fat showed a steady significant increase during aging with no appreciable differences due to curing method. Table 5 shows the mean values for all hams.

Free fatty acids developed in the seam fat at approximately the same rate as in the external fat for dry-cured hams. There were no significant differ-

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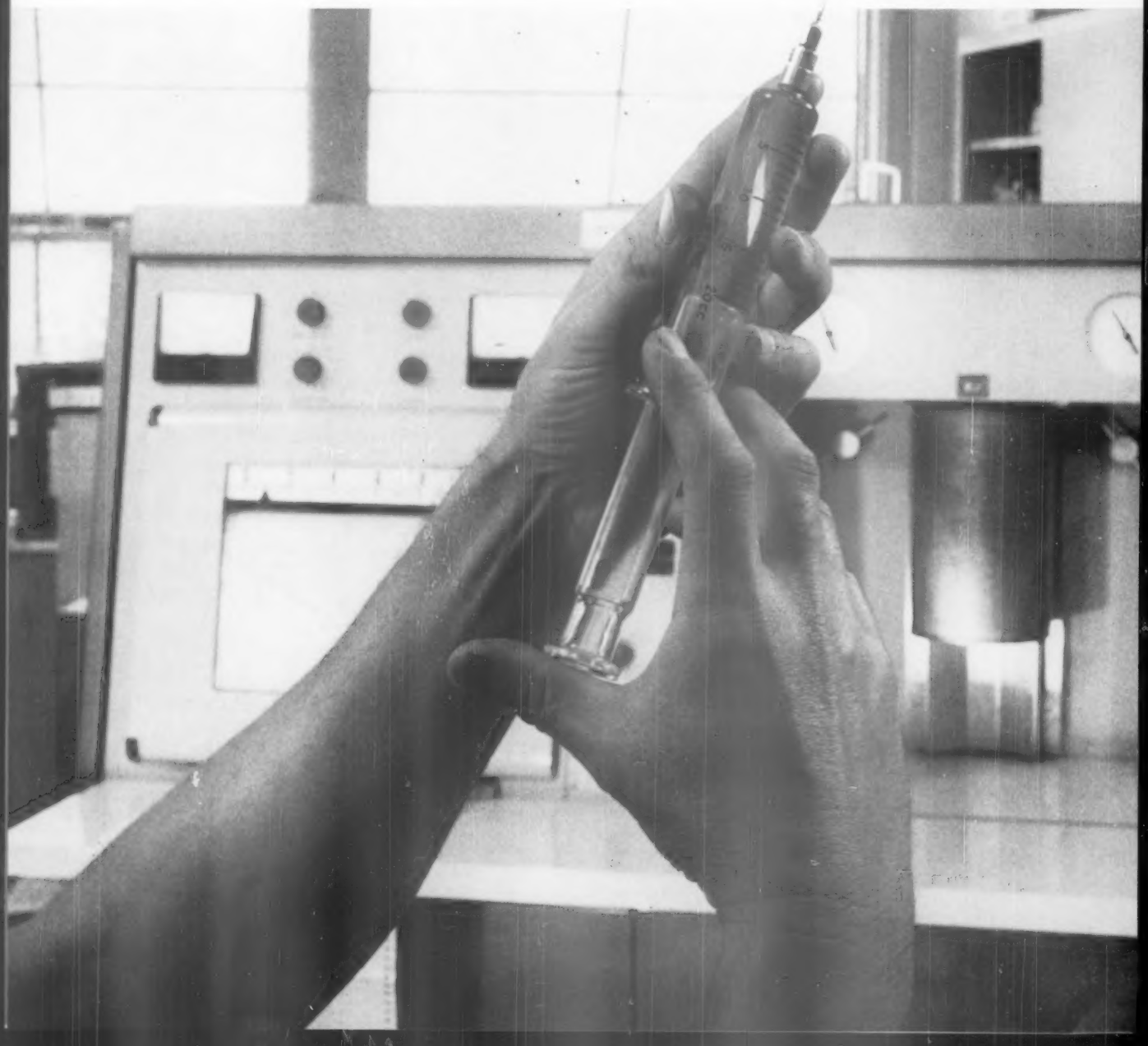

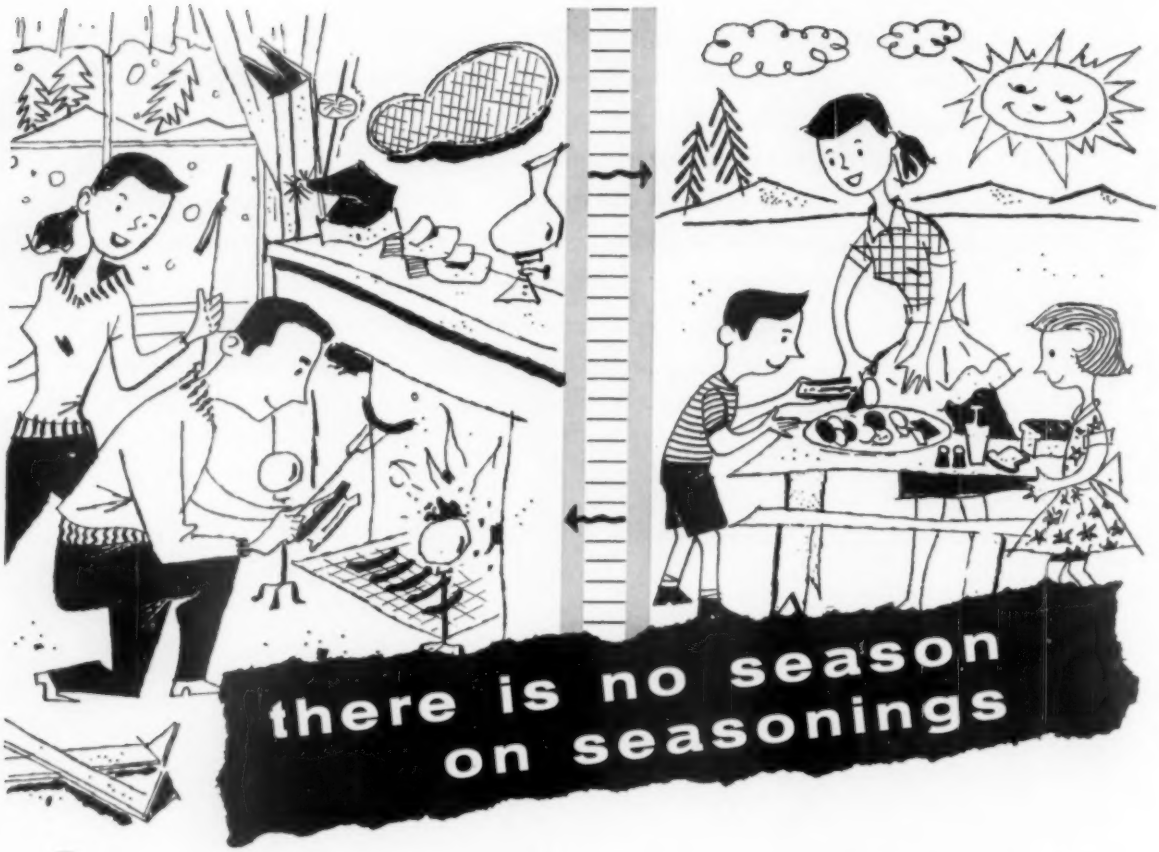

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TABLE 1

Ham shrinkage

Pumped hams—per cent shrinkage

Group No.	Pumping %	Cured	Smoked	Period of aging 1 Mo	2 Mo	3 Mo	4 Mo	5 Mo	6 Mo
1p	4	+2.57	2.79	11.08	14.75	17.77	19.78	21.55	23.01
2p	6	+4.00	1.50	9.62	13.56	16.68	18.84	20.68	22.27
3p	8	+6.10	+1.02	8.08	12.01	15.06	17.42	19.24	20.95
4p	10	+5.06	+ .33	10.27	14.37	17.62	19.95	22.03	23.80
Mean		+4.44	.36	9.76	13.67	16.78	19.00	20.87	22.51**

** Significant at .01 level between each succeeding period of aging for all groups of both pumped and dry-cured hams.

Dry-cured hams—per cent shrinkage

Group No.	Cured	Smoked	Period of aging 1 Mo	2 Mo	3 Mo	4 Mo	5 Mo	6 Mo
1d	1.55	6.72	14.94	17.59	20.21	21.88	23.41	24.62
2d	2.04	7.29	15.19	18.78	21.43	23.32	24.86	26.23
3d	2.36	7.17	15.65	19.26	21.79	23.71	25.33	26.60
4d	4.07	8.75	17.46	20.94	23.61	25.57	27.32	28.73
Mean	2.51	7.48**	15.69**	19.14**	21.76**	23.62**	25.21**	26.55**

** Significant at .01 level for all groups and periods when compared with corresponding groups and periods of pumped hams.

TABLE 2

Mean iodine values

Treatment	Fresh	Smoked	Period of aging 2 Mo	4 Mo	6 Mo
All pumped hams	63.86	63.28	64.96	63.91	64.91
All dry-cured hams	63.72	63.33	64.82	64.06	64.73

TABLE 3

Average peroxide numbers for external fat

Treatment	Fresh	Cured	Period of aging 2 Mo	4 Mo	6 Mo
All pumped hams	.95	4.06	13.07	14.48	28.08**
All dry-cured hams	1.01	4.17	15.43	17.31	25.75**

** Significant increase at .01 level for each period of aging.

TABLE 4

Mean peroxide numbers for seam fat from hams aged 6 months

Pumped			Dry-cured	
Group No	%	Peroxide No	Group No	Peroxide No
1p	4	4.98	1d	8.73
2p	6	4.94	2d	7.33**
3p	8	5.88	3d	7.26**
4p	10	7.18	4d	7.78
Mean		5.74		7.78**

** Significant at .01 level when compared with pumped hams.

TABLE 5

Mean values for free fatty acids in external fat

Treatment	Fresh	Smoked	Period of aging 2 Mo	4 Mo	6 Mo
Pumped	.25	.89	3.51	4.75	5.75**
Dry-cured	.25	.77	3.46	4.64	5.80**

** Significant at .01 level for periods of aging.

ences due to rate of pumping. Fatty acids developed significantly faster in the seam fat of pumped hams than in the paired dry-cured hams. This is shown in Table 6.

TABLE 6

Mean free fatty acid values of seam fat from hams aged 6 months

Pumped			Dry-cured	
Group No	%	F.F.A.	Group No	F.F.A.
1p	4	7.02	1d	5.40**
2p	6	8.65	2d	6.81**
3p	8	7.08	3d	7.16
4p	10	5.85	4d	4.85*
Mean		7.15		6.06**

* Significant at .05 level when compared with pumped hams.
** Significant at .01 level when compared with pumped hams.

Salt. Although there was an increase in salt content of the pumped hams with an increase in pumping rate, the differences were not significant. In all cases, the pumped hams were significantly higher in salt content than their dry-cured mates. This is shown in Table 7.

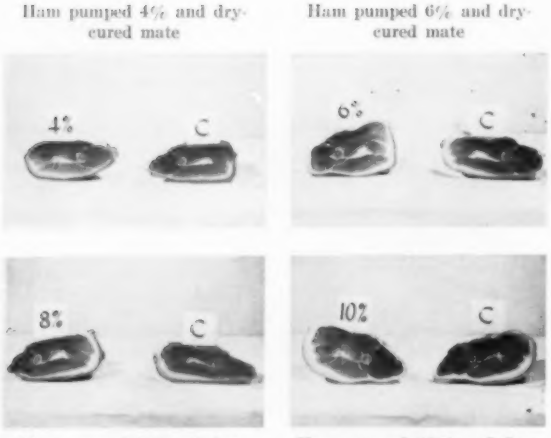
TABLE 7

Mean sodium chloride percentages

Pumped			Dry-cured	
Group No	Pumping %	NaCl%	Group No	NaCl%
1p	4	5.62	1d	4.61**
2p	6	5.60	2d	4.63**
3p	8	6.08	3d	4.77**
4p	10	6.29	4d	4.96**
Mean		5.90		4.74**

** Significant at .01 level when compared with pumped hams.

Ham quality and appearance. The hams were observed after cutting for color, general appearance, and aroma. Color ratings used were dark red, red, and light red. The aroma ratings were typically aged, moderately aged, and slightly aged. Most of the dry-cured hams were rated as red and typically aged. Group 1p of the pumped hams resembled the dry-cured hams. The other pumped groups were generally light red in color and varied in aroma scores. One ham in group 4p was sour, and several, mostly in groups 2p, 3p, and 4p showed green and yellow spots. Figure 1 shows representatives of the different groups.



Ham pumped 4% and dry-cured mate | Ham pumped 6% and dry-cured mate

Ham pumped 8% and dry-cured mate | Ham pumped 10% and dry-cured mate

Figure 1. Cross sections of paired hams.

Palatability tests. A hedonic scale scoring table was used for evaluating flavor, saltiness, tenderness, and over-all satisfaction. The highest possible score (9.0) was *like extremely*, and the lowest possible score (1.0) was *dislike extremely*. The values are given in Table 8.

TABLE 8

Mean panel palatability scores

Group No	% Pump	Flavor	Saltiness	Tenderness	Over-all satisfaction
Pumped hams					
1p	4	7.21	6.83	7.27	7.20
2p	6	6.70	7.04*	7.68	6.92
3p	8	7.21	6.78	7.40	7.12
4p	10	6.43	6.44*	7.33	6.62
Mean		6.89	6.76	7.42	7.06
Significant difference at .05 level between these groups only.					
Dry-cured hams					
1d		7.39	7.28**	7.32	7.35
2d		7.66**	7.47**	7.62	7.62**
3d		7.59	7.45**	7.31	7.49*
4d		7.49**	7.24**	7.48	7.41**
Mean		7.53**	7.36**	7.43	7.47**

* Significant at .05 level when compared with corresponding groups of pumped hams.

** Significant at .01 level when compared with corresponding groups of pumped hams.

Analysis of variance showed no significant difference in flavor, tenderness, or over-all satisfaction among the pumped hams. Saltiness was significantly less desirable, however, in group 4p than in group 2. The dry-cured hams scored significantly higher than pumped hams in flavor, saltiness, and over-all satisfaction, but curing method had no effect on tenderness. When groups 1p (4% pump) and 1d were compared, only saltiness showed a significant difference indicating that group 1p was quite similar to dry-cured hams.

SUMMARY

Sixty-four hams were divided into eight equal groups. Four groups were dry-cured, and the companion hams in four groups were partially pumped with an 85° pickle made from the dry-cure mixture to 4, 6, 8, and 10% respectively, and were than rubbed with dry-cure mixture to allow the total curing ingredients to equal the dry-cured groups. All hams were cured for 21 days at 36–40° F (2.2–4.4° C). After smoking at a smokehouse temperature of 100° F (35.6° C), the hams were aged for 6 months at a temperature of 65° F (18.3° C) and a relative humidity of 55%. Hams were weighed to the nearest 0.01 pound when fresh, after pumping, after curing, after smoking, and at monthly intervals for the 6 months aging period. Fat characteristics of external fat were determined by iodine numbers, peroxide numbers, and free fatty acid content. After 6 months aging, fatty acid content and peroxide numbers were determined for seam fat. Hams were cut and observed for color, soundness and odor. Two slices were broiled for palatability study, and a third slice was analyzed for sodium chloride.

Shrinkage increased throughout the processing and aging period, with the greatest amount occurring during the first month of aging. Pumping reduced shrinkage throughout aging. Iodine numbers were not affected by method of curing. Peroxide numbers and free fatty acid content increased during aging with no significant difference in external fat due to method of curing. Free fatty acid content was higher, and peroxide numbers were lower in seam fat of pumped hams. Salt content was higher in pumped hams with a general increase as the pumping rate increased. With the exception of hams pumped 4% dry-cured hams were sounder, showed a more desirable color, and were scored significantly higher by a palatability panel for saltiness, flavor and over-all satisfaction with no difference in tenderness. Hams pumped 4% were quite similar in quality to dry-cured hams.

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Studies on the Application of Infrared in Food Processing[a, b]

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INFRARED RADIATION as a source of heat has been studied for a number of applications in the food processing industry *(1, 10, 11, 17)*. In several cases, such as the drying of macaroni *(2)*, the roasting of coffee *(7)*, the blanching of celery and apples prior to freezing *(4)* and the peeling of apples *(3)*, infrared reportedly offered advantages as compared to other heat sources or processing techniques. These advantages differed in each particular case and varied from faster drying to improved flavor and texture. In other instances the use of infrared was not successful because of technical or economic reasons *(13, 16)*. Shuman and Staley *(16)* stated that the disappointing results obtained were often due to popular misconceptions of infrared radiation, especially with respect to its penetration characteristics. These workers suggested that an evaluation of the uses of infrared should include fundamental studies on the properties of the heat source and the absorption characteristics of the produce. A survey of the available literature revealed a lack of such fundamental data. Only in the studies on the dehydration of orange juice *(16)* and the infrared peeling of apples *(3)* were the fundamentals of infrared radiation considered and related to the specific application.

In recent years, the manufacturers of infrared equipment have produced high energy content infrared radiators (1000–5000 watts) in the form of quartz lamps *(9)*, quartz tubes *(9)* and calrods *(11)*, which can be installed with greater convenience than the old bulb type lamp of low energy content (250–1000 watts). The changes in lamp design promise wider application in many industries, including the food processing industry *(5, 15)*.

The present study was designed primarily to evaluate the three high energy, infrared radiators. The basic procedure consisted of determining the degree of heat penetration in apple tissue as effected by radiant energy produced by quartz lamps, quartz tubes and calrods. In addition, an evaluation of the following food processing applications of infrared heating is included: blanching of apples, celery and peas, the production of french fries, and the braising of stewing beef for canned stew.

EXPERIMENTAL EQUIPMENT

The 3 types of infrared radiators used in the present study are described as follows:

a. Quartz lamp, consisting of a tungsten filament, surrounded by inert gas and suspended in a sealed, translucent, fused quartz tube.

b. Quartz tube, consisting of a nichrome wire, surrounded by air in an unsealed, translucent, fused quartz tube.

c. Calrod, consisting of a nichrome coil surrounded by magnesium oxide insulation and sealed in stainless steel tubing.

The quartz lamp tunnel, Figure 1, used in the present study

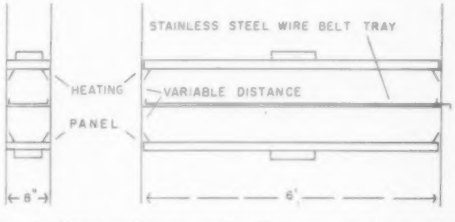

Figure 1. Plane and cross-section diagrams of the quartz lamp tunnel.

consisted of an upper and lower heating panel containing, respectively, three and two 3800 watts heating units.[c] Each lamp was 38 in long with a 4½ in wide reflector of specular aluminum. The distance between the lamp and the produce on the stainless steel wire belt was variable from 5 to 20 in. The sides of the tunnel were left open to facilitate observation and to prevent the accumulation of heat inside the tunnel.

The quartz tube tunnel was similar in design to the quartz lamp tunnel, except that each heating unit[d] was 72 in long.

The calrod heating tunnel was similar in design to the quartz tube tunnel, except that both the upper and lower panels contained one radiant panel, each equipped with two 3000 watts heating units[e] and anodized aluminum reflectors.

The relationship between input voltage, energy output, and spectral distribution for quartz lamps has been described by Barber *(5)*. For instance, at an input of 650 volts instead of at the rated 550 volts, the energy output of quartz lamps increases by approximately 26% and the location of the peak wavelength is changed from 1.15 mc to 1.06 mc; if the input voltage is changed from 550 volts to 450 volts, the energy output is reduced by approximately 30% and the peak wavelength occurs at 1.26 mc. Similarly, the energy output of quartz tubes and calrods can be varied by over- and under-voltage operation. However, for these radiators the location of the peak wavelength is much less affected because variations in the color temperature of nichrome are more limited. The color temperature of quartz tubes can be varied only between

[a] Contribution no 64, Plant Research Institute, Research Branch, Canada Department of Agriculture, Ottawa, Ontario.

[b] Presented at the Twentieth Annual Meeting, Institute of Food Technologists, San Francisco, California, May 17, 1960.

[c] General Electric, quartz lamp, type A-105-7.

[d] Quartz Products Corp., quartz infra-tube.

[e] General Electric, infrared radiant calrod panel, type QX1AA.

1000 and 1300° K; the color temperature range for calrods is between 1000 and 1150° K.

In order to study the effects of over- and under-voltage operation of the quartz lamps and quartz tubes, the wiring diagram included a transformer (5 kva), a variac (5.2 kva) and a double pole double throw switch (50 amps). In this way the input voltage of these radiators could be varied between 420 and 670 volts. Similarly, the upper heating panel of the calrod tunnel could be equipped with two 3000 watts units for in series operation at their rated 550 volts, or with two 3000 watts units for in series operation at 550 volts instead of their rated 460 volts.

The color temperature of the quartz lamps under the various conditions of voltage input was measured by means of a megatron [f] pyrometer; the color temperatures of the quartz tubes and calrods were measured with a radiation pyrometer.[g] The locations of peak wavelength were then calculated according to Wiens' displacement law.

The energy output of the upper panel of the quartz lamp, quartz tube, and calrod tunnels under the various conditions of voltage input and exposure distance was measured with a suitable thermopile and potentiometer.[h] The design, calibration and operation of this thermopile, together with data on the energy pattern in the heating tunnels, are described in a separate publication *(6)*. It should be pointed out that this thermopile measured total energy output of each radiator, regardless of the spectral distribution.

Concomitant with the energy measurements by means of the thermopile, the heat penetration into apple tissue was determined as follows: cylindrical apple sections, 1½ in long by ¾ in diameter, were cut from Fancy grade Northern Spy apples. The maturity range of the apples was 12–14 lb firmness, as measured with the Magness and Taylor pressure tester. The apple sections, which were free of core and skin tissue, were cut immediately before use. In each test 20 apple sections were placed upright in the centre of the wire belt tray. The distance between the sections was approximately ½ in. The tray was then placed in the heating tunnel and exposed to the radiation from the upper heating panel. After the selected exposure time, the apple sections were removed and cut longitudinally into two equal portions. The peroxidase activity of the cut surface was determined by applying a few drops of .1% solutions of guaiacol and hydrogen peroxide. The depth of heat penetration was defined as the thickness of the layer of peroxidase negative tissue.

RESULTS AND DISCUSSION

I. Study of energy penetration

In Figure 2 are summarized the data on heat penetration produced by quartz lamps, quartz tubes, and calrods. Results indicate that depth of heat penetration is affected by type of radiator, voltage input, and energy output. However, the interpretation of the data should be subjected to the following considerations: a. the absorption spectrum of water, b. the absorption spectrum of water vapour, c. the spectral emissivity of fused quartz.

The absorption spectrum of water, Figure 3, indicates that radiation of wavelength shorter than 1.4 mc is not absorbed by a 10 mm layer of water. Therefore, the energy of wavelengths less than 1.4 mc should be subtracted from the energy value as obtained with the thermopile.

It may be expected that the atmosphere between the radiators and the apple sections on the wire belt contains a considerable amount of water vapour due to evaporation from the apple sections. For example,

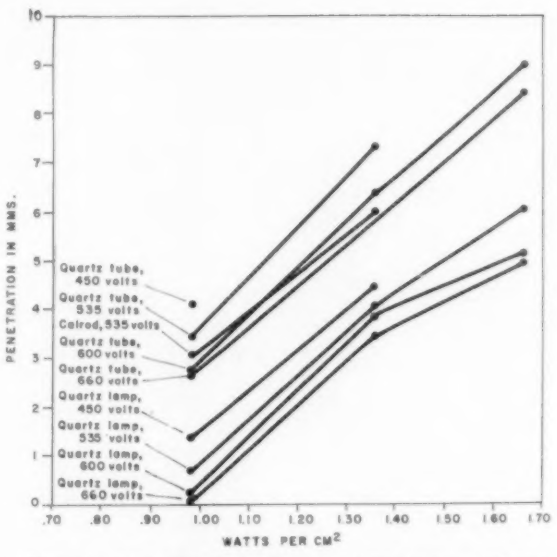

Figure 2. Heat penetration into apple tissue by three infrared sources during 5 minutes exposure at various voltage inputs.

it was found that during a 5 min exposure time at 1.36 watts per cm², the apple sections lost approximately 11% of their fresh weight. In Figure 4, it is shown that little radiation beyond 5 mc will pass through water vapour. Therefore, energy of wavelengths beyond 5 mc should be subtracted from the energy value as obtained with the thermopile.

Data compiled by Barber *(5)* on the emissivity of fused quartz show that the fused quartz envelopes do not transmit radiant energy beyond 5 mc. Nearly all energy of wavelengths beyond 5 mc is absorbed by the fused quartz envelope and is not available as radiant energy to the apple sections.

On the basis of these considerations a correction was applied to the energy levels as obtained with the thermopile. The percentage of energy between wavelengths 1.4 and 5.0 mc was calculated for each radiator. The procedure for this calculation has been described *(8)* and is based on the laws of Planck, Stefan-Boltzmann and Wien.

Based on the data presented in Figure 2, the heat penetration at a "corrected" energy level is sum-

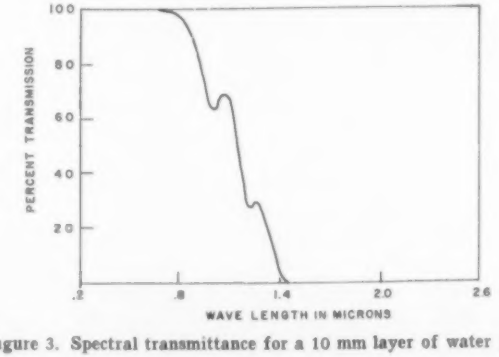

Figure 3. Spectral transmittance for a 10 mm layer of water *(9)*.

[f] Megatron Corporation.

[g] Leeds and Northrop.

[h] Rubicon Company, Cat. No. 2745.

TABLE 1

Heat penetration into apple tissue after 5 minutes exposure to three types of infrared radiators at voltage input of 535 volts

Radiator	Energy as per thermopile	λ max	"Corrected" energy between 1.4 and 5.0 microns		Penetration	Standard error
	watts/cm²	*microns*	*per cent*	*watts/cm²*	*mm*	*mm*
Quartz lamp	1.36	1.16	56.5	.77	4.1	.068
Quartz tube	1.36	2.35	72.0	.98	7.4	.110
Calrod	1.36	2.65	67.0	.90	5.9	.186

marized in Table 1. The data indicate that the depth of penetration corresponds closely to the amount of energy available for each radiator between wavelengths 1.4 and 5.0 mc.

From the data for quartz lamps and quartz tubes (Figure 2) it appeared that the depth of penetration decreased with increased voltage input. In Table 2 are presented the amounts of "corrected" energy required at various voltage inputs to produce 4 mm penetration into apple tissue. Results indicate that at a voltage input of 535 volts, the energy outputs required on the basis of thermopile data would be 1.35, 1.03 and 1.10 watts per cm² for the quartz lamp, quartz tube and calrod, respectively. However, on the basis of "corrected" energy levels these figures change to .75, .75 and .74 watts per cm.² Furthermore, the amount of "corrected" energy required from quartz lamps for 4 mm penetration decreases with increased voltage inputs. In the case of quartz tubes the amount of "corrected" energy required increases with increased voltage inputs. In other words, the efficiency of quartz lamps increases with increased voltage input and the efficiency of quartz tubes decreases with increased voltage input.

It may be noticed in the present study that the infrared absorption spectrum of apple tissue has not been considered. The infrared absorption characteristics of apple solids become important only during the advanced stages of dehydration and do not apply to experiments where short exposure times are used.

II. Application to products

a. *Blanching of celery.* The harvesting season of celery in Ontario is limited to a 6-week period during September and early October. This period coincides

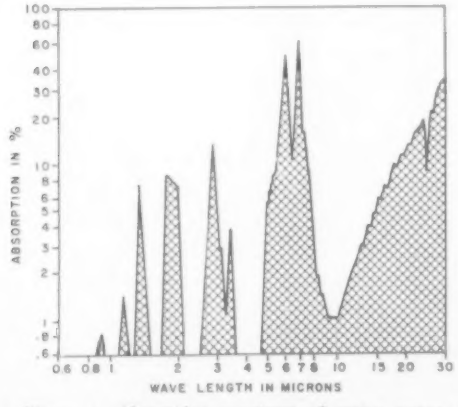

Figure 4. Absorption spectrum of water vapor *(9).*

TABLE 2

Effect of type of radiator and voltage input on the amount of energy required to produce 4 mm heat penetration in apple tissue

Radiator	Voltage input	λ max	Required energy as per thermopile	"Corrected" energy required between 1.4 and 5.0 microns	
	volts	*microns*	*watts/cm²*	*per cent*	*watts/cm²*
Quartz lamp	450	1.26	1.30	62.0	.81
	535	1.16	1.35	56.5	.75
	600	1.12	1.39	53.5	.74
	660	1.06	1.46	50.0	.73
Quartz tube	535	2.35	1.03	72.0	.74
	600	2.26	1.11	73.0	.81
	660	2.10	1.14	75.0	.86
Calrod	535	2.65	1.10	67.0	.74

with optimum canning activity for tomatoes and therefore the production of celery soup is delayed until the winter months. However, celery cannot be stored for more than 30 days in cold storage (38° F) and in order to have a supply of celery for soup stock, the celery is frozen during the harvest season for remanufacture at a later date. If celery is not adequately blanched before freezing a bitter flavor develops during storage. The customary procedure is to trim and cut celery stalks into ½ in pieces, followed by a steam or boiling water treatment of 3 to 4 min. The product is then packed in 30 lb bags and frozen immediately.

In preliminary experiments it was noticed that the cut celery, after exposure to infrared for 3 to 6 min, would be peroxidase negative except for the outer .5 mm layer of surface tissue. The peroxidase activity in this region could be eliminated by dipping the celery in boiling water for 15 seconds prior to exposure to the infrared. The procedure adopted for infrared blanching of celery is briefly as follows: celery stalks, free of leafy tissue, were cut in ½ to 6 in lengths, dipped for 15 seconds in boiling water, exposed to infrared radiation from quartz lamps for 2 to 3 min, packaged in polyethylene bags and frozen immediately. The wattage requirement from the upper and lower heating panels was approximately 1.50 watts per cm² per panel, as measured with the thermopile. Accompanying weight losses of the celery were between 25 and 35%. Load density of the fresh produce was approximately 1 lb per sq ft of belt.

After freezing and storage at 0° F for 3 and 6 months, samples of infrared- and water-blanched celery were evaluated organoleptically. The infrared-blanched celery was considered superior in flavor and texture and equal in appearance as compared with the water-blanched product. One Canadian firm has used the infrared procedure on a commercial scale and in

1958 and 1959 more than 1 million lb of celery were prepared in less than 8 weeks.

b. *Blanching of apples.* Steam or vacuum blanching of apple slices results in a considerable loss of apple flavor. In an attempt to improve the flavor and general quality, infrared blanching was studied for the varieties Northern Spy, Sandow, Cortland and MacIntosh. As in the case of celery, a short dip in boiling water was necessary to inactivate peroxidase in the surface layer, especially around the core tissue. All varieties, when sliced in 8 or 10 sections, required a 4 to 5 min exposure from quartz lamps. The wattage requirement from the upper and lower heating panels was approximately 1.20 watts per cm^2 per panel, as measured with the thermopile. Weight loss of the apple slices during the blanching treatment was between 10 and 15%. The load density of produce was approximately 2.3 lb per sq ft of belt.

The color of the white-fleshed varieties, Cortland and MacIntosh, was improved if the slices were dipped for 15 seconds in 2000 ppm sodium metabisulfite prior to exposure to infrared. The yellow varieties did not require this pretreatment. After blanching, apple slices were both frozen and canned. After freezing and storage for 6 months at 0° F the frozen slices were evaluated organoleptically. The infrared-blanched slices retained better texture, flavor and color than the steam- or vacuum-blanched slices. The canning of infrared-blanched apple slices is more complicated because the tissue air is not removed during blanching. However, samples were prepared using a 15% sugar syrup and .3% ascorbic acid in both tin and glass containers. The samples pretreated with the sulphite dip and processed in tin cans (20 oz, apple juice enamel) showed corrosion of the can lining within 3 months storage at room temperature. The varieties Northern Spy and Sandow produced a very acceptable product after processing and storage at room temperature for at least 6 months. All the samples in the glass containers (16 oz, screw cap) maintained excellent flavor and texture after processing and storage at room temperature for 6 months. However, the slices in the glass containers showed darkening when exposed to sun light.

c. *Blanching of peas.* Frozen and canned samples of steam-blanched peas were compared with similar samples after blanching with infrared. Prior to exposure to infrared the peas were dipped in boiling water for 30 seconds in order to eliminate peroxidase activity in the surface tissue. The blanching required approximately 4 min exposure to infrared radiation from quartz lamps. The wattage required from the upper and lower heating panels was approximately 1.30 watts per cm^2 per panel, as measured with the thermopile. Accompanying weight losses of the peas were between 25 and 35%. The load density of fresh peas was approximately 1.1 lb per sq ft of belt.

Organoleptic evaluation of heated samples of frozen peas after 5 months of storage at 0° F showed equal quality for the steam-blanched and infrared-blanched peas. Canned samples also were similar in quality, but the infrared-blanched peas had a more pronounced pea flavor. Because of shrinkage of the peas during the infrared treatment, more peas could be filled into the tin container. After processing and storage for 5 months at room temperature the drained weight of infrared-blanched peas was 13% higher than that of the steam-blanched peas.

d. *The production of french fries.* Procedures for the preparation of french fries by means of deep fat frying, infrared radiation, and a combination of both are described in a separate publication *(12)*. Oil-blanching of french fries with infrared, as compared to conventional deep fat frying, resulted in 8 to 12% and 4 to 8% higher product yields after the first and second stage preparations, respectively. In addition, a 2 to 4% lower fat content in the stage one and stage two product was obtained. When the parfries were frozen and stored at 0° F between stages one and two, the finished product was of only fair quality, regardless of the technique used to prepare the first stage product. Parfries stored up to 9 days at 38° F between stage one (infrared) and stage two (deep fat fry) resulted in a finished product of good quality.

e. *Braising of meat for beef stew.* Preparing large quantities of meat for beef stew in steam-jacketed kettles results in a "cooked" meat appearance and weight-losses up to 35%. This method of preparation is also time consuming, especially if frozen meat is used. The parboiling treatment used in this study consisted of boiling one volume of frozen meat (1 x 1 x 1 in cubes of lean round sirloin) with 2 volumes of water containing 2% sodium chloride, for approximately 6 min. This period was sufficient to cook each meat cube completely. It was considered that this condition was reached when the inside of the meat cube showed no residual red color. For a similar degree of cook the infrared treatment required an exposure time of 5 min from quartz tubes. The wattage requirement from the upper and lower heating panels was approximately 2.20 watts per cm^2 per panel, as measured with the thermopile. The load of meat per square foot of belt space was approximately 1.9 lb. Weight losses during the parboiling and infrared treatments were, respectively, 30 and 15%. The flavor, color and texture of the infrared-braised meat was much superior to the parboiled meat. When the period of cook for the parboiled and infrared treatment was reduced to 50% residual inside red color, the corresponding weight losses became 25% and 10%, but the outside appearances of the samples did not change.

Measured quantities (approximately 4 oz) of parboiled and infrared treated meat were added to 20 oz cans, containing approximately 16 oz of beef stew prepared with a standard formula. After processing (110 min at 240° F) the cans were stored at room temperature. Analysis of the canned samples after a 6-week storage period indicated that during the retorting the weight losses of the parboiled and infrared prepared meat were approximately 5 and 20%, respectively. This indicates that the total weight losses of the meat after the complete canning operation was

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approximately equal for the parboiled and infrared techniques.

f. *Blanching of asparagus, corn, beans, turnips and carrots.* Preliminary attempts to blanch asparagus, whole kernel corn, corn-on-the-cob, cut green beans, diced turnips and diced carrots were unsuccessful. No combination of type of radiator, exposure distances and exposure period would produce sufficient heat penetration to inactivate peroxidase. In most cases the exposure to infrared resulted in surface scorching before heat penetration was more than a few millimeters.

SUMMARY

Three types of high energy, infrared radiators—quartz lamps, quartz tubes, and calrods—were evaluated for their effectiveness in blanching apple tissue. Data are presented which show how the depth of heat penetration was influenced by wavelength characteristics, voltage input, and energy output of the radiator.

Infrared radiation was used successfully for blanching celery, apples, and peas prior to freezing and in the preparation of french fried potatoes. General quality of infrared-blanched samples was better than that of samples blanched by conventional steam or boiling water methods. Accompanying weight losses of up to 35% by weight would result in reduced freezing, storing, and handling costs in the case of products intended for later remanufacture.

Infrared was substituted for the par-boiling treatment of beef as the preliminary heat treatment in the preparation of beef stew. The colour, texture, and flavour of the infrared-braised, canned meat was considered superior to that of the par-boiled meat.

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Comparative Heating Efficiencies of a Microwave and a Conventional Electric Oven[a]

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(Manuscript received May 18, 1960)

To BOTH the commercial and military user, the efficiency with which input electrical energy is converted to heat energy usable for cooking food, is of economic importance. Microwave cooking is of comparatively recent origin, having been developed within the past 15 years. In reviewing the status of microwave cooking in 1947, Brown, Hoyler, and Bierwirth *(1)* commented, among other aspects, on the low efficiency of microwave cooking. Sussman *(4)* subsequently reported that the use of a microwave cooker "required 56% less electrical energy than an electric range for producing a given quantity of heat, such as evaporating a given quantity of water to dryness." However, in producing a complete meal for 10 men, Sussman found that the energy requirements were greater for the microwave oven than for the electric range. No further studies of the heating efficiency of microwave equipment were reported in the literature, but later investigators *(2)* imputed a high efficiency to microwave cooking based primarily upon the assumption that the extremely short cooking time of microwave equipment must use less electrical energy than the longer cooking times of conventional equipment.

The purpose of the present study was to determine heating efficiencies in both microwave and conventional equipment for a typical meat item, roast beef, and for the heating of water because water is the medium used to cook most vegetables. The work was part of a study on the applicability of microwave cooking to Navy subsistence.

EXPERIMENTAL METHODS

Equipment and instrumentation. The cooking equipment used has been described by Pollak and Foin *(3)*. The microwave oven was equipped with one Type QK-390 magnetron operating at 2,450 megacycles, a wave-dispersing fan, and an auxiliary browning element. The conventional oven was a modern, heavy-duty, Navy electric range, designated as the Navy Type 8 Range in accordance with military specification MIL-R-21211 (SHIPS). Temperatures were measured with an assembly of twelve thermocouples and recorded on high-speed strip chart potentiometers. Continuous recordings were taken for the operations with the conventional electric range. However, since thermocouples cannot be used during operation of the microwave cooker, thermocouple readings were taken immediately prior to and after microwave operation. During microwave operation, temperature was monitored by visual observation of a special thermometer, Taylor Instrument Co. Model No. Z 4505. Power demand was recorded continuously on strip-chart recorders and total energy was recorded by integrating wattmeters. Both microwave and conventional equipment were brought to temperature equilibrium or normal operating condition prior to any test.

Procedures. *Heating water.* After measuring temperature, a fixed weight of water in a tempered glass bowl with cover was heated in the microwave and conventional oven and on a hot plate of the conventional range to a temperature of 200° F. The conventional oven was operated at 450° F, the conventional hot plate at 750° F, and the microwave oven at maximum power input.

Cooking beef. Standing short ribs of beef, U. S. Grade B, 9th to 11th rib, of uniform weight, size, shape, fat and bone configuration, were prepared, one piece at a time, in each oven. In the microwave oven, the meat was cooked in a tempered glass dish, at maximum power, fat-side down, for about half the cooking time and was then inverted for the remaining time. In the conventional oven, the meat was roasted, rib-side down, at 325° F, using "high" top and bottom heat until temperature recovery was attained; the bottom heat was then reduced to "medium" and the top heat was turned off for the remainder of the roasting period. In both types of cooking equipment, the meat was timed to attain an internal temperature, at the center of the meat, of about 145° F.

RESULTS AND DISCUSSION

Heating water. The experimental data for heating of water are given in Table 1. The efficiencies for the microwave and conventional oven are not significantly different, both being about 34%. The conventional hot plate, on the other hand, had an efficiency of about 63% or nearly double that of the ovens. These results are not unusual when the construction of the equipment is considered. In the hot plate, the heating element is in direct physical contact with the surface plate which is in direct contact with the cooking container permitting efficient heat conduction. This permits much better heat transfer than that which takes place in the oven, where large masses of metal and air must be heated and about half the heat input (from top elements) must be transmitted to the product solely by radiation and convection.

Roasting beef. The experimental data for the preparation of beef are given in Table 2. The efficiency of the conventional oven, 36.7%, is slightly higher than and significantly different (at the 5% level) from that of the microwave oven, 33.4%. This similarity in efficiency, however, does not mean that the usable heat energy performs the same job in both types of cooking. In terms of edible meat resulting from the cooking process, the conventional oven yields 13% more edible meat. Furthermore, it does so using 24% less total input electrical energy. The conventional oven is therefore doing a better job, but some means other than electrical efficiency must be used to

[a] Presented at the Twentieth Annual Meeting of the Institute of Food Technologists, San Francisco, California, May 17, 1960.

[b] Present address: The A. L. Mathias Company, Baltimore, Maryland.

TABLE 1

Experimental data used to compute comparative electrical efficiencies of microwave oven and conventional electric range for heating water

Equipment used	Microwave oven				Conventional oven				Conventional hot plate			
Test number	1	2	3	4	5	6	7	8	9	10	11	12
Weight of glass bowl and cover, oz	60	60	62	60	62	60	62	60	60	62	60	62
Weight of water, oz	80	80	80	80	80	80	80	80	80	80	80	80
Water temperature, initial, ° F	71	71	72	72	70	69	70	70	70	70	71	70
Water temperature, final, ° F	198	191	194	198	200	200	200	200	200	200	200	200
Total heating time, minutes	13	13	13	13	32.7	31.5	30.3	28.2	11.5	10.3	10.4	10.5
Electrical "on" time, minutes	13	13	13	13	7.5	8.1	8.1	7.8	11.5	10.3	10.4	10.5
Power demand, kilowatts	2.85	2.81	2.80	2.85	4.9	5.0	5.1	5.0	1.95	1.95	1.95	2.0
Total energy, kilowatt-hours	0.62	0.61	0.61	0.62	0.61	0.68	0.69	0.65	0.37	0.33	0.34	0.35
Efficiency, per cent	34.5	33.1	33.8	34.2	36.0	32.4	31.9	33.7	59.2	66.6	63.9	62.8
Average efficiency, per cent	33.9				33.5				63.1			

express this situation. A computation was made of the energy (BTU) input per ounce of yield and this resulted in values of *74 BTU per ounce of yield* for the conventional oven compared to *119 BTU per ounce of yield* for the microwave oven. Further examination of the experimental data reveals that the largest difference in energy outputs was that required to evaporate moisture losses from the meats. For the conventional oven, only 60% of the total output energy was required to evaporate moisture losses as compared to 76% for the microwave oven. Thus, it can be seen that cooking in the microwave oven dried out the meat more than in the conventional oven. This confirms observations made by the authors *(3)* in yield studies. As a result, the difference in the energy required to prepare meat by the two methods is attributed directly to the amount of moisture evaporated during cooking.

Another aspect of the cooking process which can be explained by this study is the relation between cooking time and energy input. Microwave cooking is very fast. In this study, beef was prepared by microwave equipment in 34% of the time required in conventional equipment. Also, the instantaneous power demand was only 56% that of the conventional oven. However, power is on for 100% of the cooking time in the microwave oven as compared to cycling under thermostatic control in the conventional oven. In the conventional oven, power was on for only 2% of the time on "high" heat (4 minutes at 5.0 kw) and for only 30% of the time on "medium" heat (55 minutes at 2.0 kw). From these figures, it can be seen why the total energy output to do the same cooking job is less for the conventional oven than for the microwave oven, under the particular conditions of this study.

METHODS USED FOR CALCULATING EFFICIENCY

Heating of water. The efficiency of electrical energy conversion for heating water was calculated using equation (1) from the data in Table 1:

$$\% \text{ Efficiency} = \frac{(T_2 - T_1)(M_w) + (T_4 - T_3)(M_g)(S_g)}{E\ (3415)} \times 100 \qquad (1)$$

where:

T_1 = Initial temperature of water, ° F
T_2 = Final temperature of water, ° F
M_w = Weight of water, lb
T_3 = Initial temperature of glass container, ° F
T_4 = Final temperature of glass container, ° F
M_g = Weight of glass container, including cover, lb
S_g = Specific heat of glass container
E = Input energy, kwh
3415 = BTU per kwh

Roasting of beef. The total energy inputs were obtained directly from the recording instruments and were converted into BTU's. The total energy outputs consist of the sum of the energy required to perform these four functions: (1) to evaporate water from the meat, (2) to heat the cooking utensil, (3) to render and heat the fat which comprised the drippings, (4) to heat the meat exeluding its drippings and moisture losses. The calculation of each of these is described below.

Energy to evaporate water from meat. By subtracting the weight of rendered liquid from the weight of total losses, the weight of fluid which was vaporized was obtained. The energy required to heat this water to vaporization temperature and then to evaporate the water was calculated using equations (2), (3), and (4):

$$Q_T = Q_s + Q_L \qquad (2)$$
$$Q_s = M_w \times (T_2 - T_1) \qquad (3)$$
$$Q_L = M_w \times h_{fg} \qquad (4)$$

where:

Q_T = Total energy, BTU
Q_s = Sensible heat energy, BTU
Q_L = Latent heat energy, BTU
T_2 = Final temperature (in this case, the vaporization temperature of water, taken as 212° F)
T_1 = Initial temperature (taken same as temperature of meat before cooking)
M_w = Weight of water
h_{fg} = Heat of vaporization at boiling point, BTU/lb

Energy to heat the cooking utensil. This was calculated using equation (5):

$$Q_s = M \times C_p \times (T_2 - T_1) \qquad (5)$$

where:

Q_s = Specific heat energy, BTU
M = Weight of glass cooking utensil, lb
C_p = Specific heat of glass pan, BTU/lb
T_1 = Initial temperature of glass pan
T_2 = Final temperature of glass pan

The initial temperature of the glass pan was 81° F during the experiments; the final temperature was taken as identical to that of the rendered fat. The specific heat of the tempered glass was taken as 0.20.

Energy to render and heat fat in pan. This was calculated using equation (6). The right-hand part of the equation takes into account the change of state of the fat from a solid in the meat to a fluid when rendered by the heat of the cooking process. It was assumed, based upon the experimentally measured temperatures of the fluid in the pan, that the rendered fluid is entirely fat:

$$Q_T = (M_w)(C_p)(T_2 - T_1) + (M_w)(h_{sf}) \qquad (6)$$

where:

Q_T = Total energy to render and heat fat, BTU

TABLE 2

Experimental data used to compute comparative electrical efficiencies of microwave oven and conventional electric oven for roasting beef

Equipment used	Microwave oven									
Test number	1	2	3	4	5	6	7	8	9	Average
Dimensions of roast										
Length, in	7½	7⅝	7	5¾	8½	7½	6½	7½	7¼	7¼
Width, in	8¾	8	9½	8¼	9½	8¾	9½	9½	9½	9
Height, in	4¼	4⅝	4½	5½	4½	4½	6	4¾	4½	4¾
Weight of pan, oz	62	64	62	64	64	64	62	64	62	63
Weight of meat before cooking, oz	130	131	126	124	137	132	130	130	128	130
Weight of meat after cooking, oz	83	96	72	69	85	87	83	86	79	82
Weight of rendered fluid, oz	5	10	19	18	15	12	12	9	13	13
Temperature of rendered fluid, ° F	225	217	222	225	225	221	222	224	223	223
Weight of total cooking losses, oz	47	35	54	55	52	45	47	44	49	48
Percentage total losses, %	36	27	43	44	38	34	36	34	38	37
Weight of liquid evaporated, oz	42	25	35	37	37	33	35	35	36	35
Temperature before cooking, ° F	62	46	49	32	76	63	51	49	48	53
Temprature after cooking, ° F										
At center of meat	162	160	143	152	123	115	138	127	144	140
Midway between surface and center	209	200	145	190	198	154	....	189	196	185
One-quarter way below surface	213	209	153	214	215	210	214	211	215	206
Cooking time, min	71	60	56	65	60	60	60	60	65	62
Power demand, KW										
High heat	2.80	2.80	2.70	2.80	2.75	2.70	2.80	2.80	2.80	2.80
Medium heat*	....	....	....	....	....	....	....	....	....	....
Power "on time", min										
High heat	71	60	56	65	60	60	60	60	65	62
Medium heat*	....	....	....	....	....	....	....	....	....	....
Total energy input, KWH	3.31	2.80	2.52	3.03	2.75	2.70	2.80	2.80	3.03	2.86
Total energy input, BTU	11,304	9,562	8,626	10,347	9,391	9,221	9,562	9,562	10,347	9,769
Energy output, BTU										
To heat and evaporate moisture	2,945	1,772	2,481	2,656	2,554	2,305	2,476	2,481	2,551	2,469
To heat glass pan	112	115	112	115	115	115	112	115	112	114
To render and heat fat in pan	41	83	160	163	115	95	100	76	110	105
To heat meat	584	720	412	569	530	538	....	629	596	572
Total energy output, BTU	3,682	2,690	3,165	3,503	3,314	3,053	....	3,301	3,369	3,260
Efficiency = Output / Input = %	32.6	28.1	36.7	33.9	35.3	33.1	....	34.5	32.6	33.4

Equipment used	Conventional oven							
Test number	1	2	3	4	5	6	7	Average
Dimensions of roast								
Length, in	7⅛	7½	7¾	6¾	10	7¼	7¾	7⅞
Width, in	8½	8½	8½	8½	6¼	8¼	8½	8¼
Height, in	4½	4½	4¾	5	5⅝	5½	4¾	5
Weight of pan, oz	62	64	62	62	62	64	64	63
Weight of meat before cooking, oz	136	124	128	137	129	131	126	130
Weight of meat after cooking, oz	96	85	100	100	103	106	104	99
Weight of rendered fluid, oz	6	11	8	10	7	7	6	8
Temperature of rendered fluid, ° F	336	314	278	320	304	301	322	311
Weight of total cooking losses, oz	40	39	28	37	26	25	22	31
Percentage total losses, %	29	32	22	27	20	19	17	24
Weight of liquid evaporated, oz	34	28	20	27	19	18	16	23
Temperature before cooking, ° F	46	51	51	57	49	47	53	51
Temprature after cooking, ° F								
At center of meat	150	151	150	150	145	142	145	148
Midway between surface and cente	184	179	183	193	188	180	181	184
One-quarter way below surface	198	201	211	214	199	210	213	207
Cooking time, min	202	185	186	187	177	175	172	183
Power demand, KW								
High heat	5.00	5.00	4.90	5.20	5.00	4.90	5.10	5.00
Medium heat*	2.00	2.00	2.00	2.00	2.00	1.90	2.00	2.00
Power "on time", min								
High heat	8.80	2.50	1.60	3.70	5.10	3.10	2.90	4.00
Medium heat*	52	61	54	60	51	57	53	55
Total energy input, KWH	2.47	2.24	1.93	2.32	2.13	2.06	2.01	2.17
Total energy input, BTU	8,435	7,650	6,591	7,923	7,274	7,035	6,864	7,396
Energy output, BTU								
To heat and evaporate moisture	2,414	1,962	1,413	1,899	1,346	1,277	1,129	1,634
To heat glass pan	198	186	153	185	173	176	193	181
To render and heat fat in pan	72	123	81	112	77	77	68	87
To heat meat	837	697	797	839	845	889	882	827
Total energy output, BTU	3,521	2,968	2,444	3,035	2,441	2,419	2,272	2,729
Efficiency = Output / Input = %	41.7	38.8	37.6	38.3	33.6	34.4	33.1	36.7

* These figures refer to the use of bottom heat on the conventional oven.

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6. RELIABILITY OF SUPPLY
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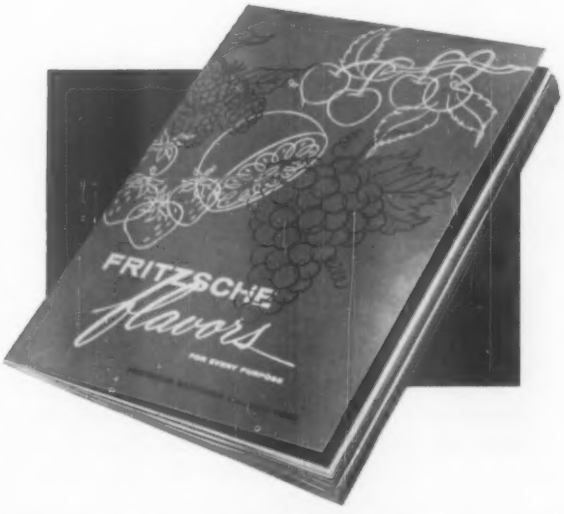

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M_w = Weight of rendered fat, lb
C_p = Specific heat of beef fat, BTU/lb
T_2 = Final temperature of fat
T_1 = Initial temperature of fat
h_{sf} = Heat of solidification of beef fat = Heat of liquefaction, BTU/lb

The specific heat of beef fat was taken as 0.50. The initial temperature of the fat was taken as the experimentally determined initial temperature of the meat immediately prior to roasting. The final temperature of the fat was determined experimentally. The heat of solidification of beef fat was taken as 48 BTU/lb.

Energy to heat meat. This was calculated from equation (7):

$$Q_s = M_w \times C_p \times (T_2 - T_1) \qquad (7)$$

where:

Q_s = Total energy to heat meat, BTU
M_w = Weight of meat, lb
C_p = Specific heat of meat, BTU/lb
T_2 = Final temperature of meat, ° F
T_1 = Initial temperature of meat

In making these calculations, the weight of meat was taken as the weight of meat after cooking since the energy relationships for the moisture and fat content have been computed separately. The specific heat of the meat was taken as 0.75 BTU/lb. The initial temperature of the meat was determined experimentally, and was uniform throughout the meat. However, it is necessary to calculate an average final temperature for the meat because the meat is not at a uniform temperature at the completion of the cooking process. The average final temperature was calculated from experimentally determined values for the temperature of various zones of the meat. This was done by calculating an average temperature for each zone and from these individual values, calculating a weighted average for the entire piece of meat, as in equation (8):

$$T_A = \frac{(T_1)(V_1) + (T_2)(V_2) + (T_3)(V_3) + (T_4)(V_4)}{V_T} \qquad (8)$$

where:

T_A = Average temperature for entire piece of meat
T_1 = Average temperature of zone corresponding to V_1
V_1 = Volume of outermost zone

T_2, T_3, and T_4 are average temperatures of zones V_2, V_3, and V_4. The zones selected were bounded by concentric spheroids, except that T_4 was the center spheroid. The average temperature of each zone was taken as the average of the temperatures of the inner and outer surfaces of each zone, except for the center spheroid, where the center temperature was taken as the temperature of the center spheroid. The magnitude of error here is small because the central spheroid comprised less than 2% of the total volume.

Efficiency. The sum of the four factors described above represents total energy output. Total energy input was measured directly by recording instruments. The ratio of total energy output to total energy input was calculated as efficiency.

SUMMARY

A study was made of the heating efficiency of microwave and conventional cooking equipment in the heating of water and the roasting of beef. In heating water, the overall electrical efficiency was 34% for both microwave and conventional ovens, but the efficiency of a conventional hot plate in heating water was 63%, virtually double that of the ovens. In roasting 8-pound ribs of beef, the overall electrical efficiencies were 36.7% for the conventional oven and 33.4% for the microwave oxen. However, the product prepared in the conventional oven yielded 13% more edible meat using 24% less electrical energy. A better way of expressing the energy-yield relation is the energy required to prepare a unit weight of edible product. This was found to be *74 BTU per ounce of yield* for the conventional oven as compared to *119 BTU per ounce of yield* for the microwave oven. The difference in energy was attributed primarily to the energy required to evaporate a greater amount of water in microwave cooking.

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Comparison of Rate of Cooking and Doneness of Fresh-Unfrozen and Frozen, Defrosted Turkey Hens [a, b]

(Manuscript received May 16, 1960)

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LOWE *(3)* reported that the rate at which meat cooks is affected by such factors as the initial temperature and composition of the meat and oven temperature for roasting. The rate of cooking also may be dependent on such treatments as freezing prior to roasting. Marsden, Alexander, Schopmeyer, and Lamb *(4)* reported that fresh, chilled, not frozen Beltsville Small White and Broad Breasted Bronze turkey hens and toms required longer cooking times (min/lb) to roast than did frozen, defrosted turkeys studied in an earlier investigation *(1)*. They suggested that this difference in rate of cooking might be attributable to the freezing and the defrosting of the turkeys prior to roasting. In both studies the oven temperature was adjusted to the weight of the turkeys and varied from 262° to 326° F. Presently, 325° F *(3)* is recommended as the roasting temperature for turkeys of all weight classes. The initial temperature or range in initial temperatures of the birds was not reported *(1, 4)*.

In the study reported here, the rate of cooking and degree of doneness of fresh-unfrozen (fresh chilled) and frozen defrosted turkey hens with similar initial internal temperature and oven temperature were investigated. Also, turkeys roasted breast-up were compared with those cooked breast-down.

EXPERIMENTAL PROCEDURE

Thompson White turkey hens grown and processed under similar production and management procedures, ranging in age from 24 to 28 weeks were purchased from a local turkey grower. All turkeys were purchased fresh-unfrozen and either stored in the refrigerator (34° F) 1 to 6 days prior to roasting or wrapped in aluminum foil (0.0015 gauge), frozen, and stored (1 to 6 days) at 0° F. The turkeys roasted at a given period always were from the same lot and usually were obtained on the Saturday preceding the week that the birds were roasted.

Fresh-unfrozen vs frozen. Twenty fresh-unfrozen and 20 fresh-frozen turkeys were roasted in Experiment I. Fresh-unfrozen and fresh-frozen turkeys were roasted to internal temperatures of 90° C in the right breast *(pectoralis major)* and to 95° C in the right thigh muscles. The turkeys were roasted, 4 at a time, according to a randomized complete block design. A block consisted of 2 fresh-unfrozen and 2 fresh-frozen turkeys each roasted to either 90° C in the breast or 95° C in the thigh.

Forty turkeys were roasted in Experiment II. Of these, 12 fresh-unfrozen were compared with 12 fresh-frozen turkeys. In addition, 8 were roasted after 1 mo storage and 8 following 3 mo at 0° F. These turkeys were roasted 2 at a time according to a randomized complete block design. For the fresh-unfrozen vs the fresh-frozen turkeys, a block consisted of 1 fresh-unfrozen and 1 fresh-frozen turkey. Four cooking periods were used for turkeys stored for 1 mo and also for those stored 3 mo. All turkeys in Experiment II were roasted to an internal temperature of 95° C in the right thigh. Weights of the turkeys ranged between 10.4 and 10.8 lb for the 2 years of the study.

Breast-up vs breast-down. Also, 18 fresh-frozen turkeys were roasted in Experiment III. Nine of these birds were cooked breast-up during the entire roasting period, and 9 were cooked breast-down for the first hour and then turned breast-up for the remainder of the roasting period. These turkeys were roasted 2 at a time according to a randomized complete block design. A block consisted of 1 turkey roasted breast-up and 1 turkey roasted breast-down. These turkeys also were roasted to an internal temperature of 95° C in the right thigh. Weights for these turkeys ranged between 9.4 and 10.1 lb.

Defrosting and roasting. Wrapped frozen turkeys were defrosted at room temperature (80° F) for approximately 18 hr prior to roasting in a rotary hearth gas oven maintained at 325° F. All turkeys were roasted on a V-rack in an open pan with the breast-up unless otherwise indicated. In preparation for roasting, skin was pulled over the openings at the neck and posterior of the bird and fastened by sewing to prevent excess moisture loss from the body cavity.

Thermometers were inserted midway between the medial and lateral side at the center of the *pectoralis major* and the thigh muscles. Thermometer bulbs were midway between the dorsal and ventral sides of the muscles. The initial temperature of the muscle in which the end point was taken was similar for all turkeys studied the same year. Internal temperatures were recorded (Experiments II and III) after reading thermometers through an oven door window. Time required for each 15° C rise in internal temperature was recorded until 60° C was reached, and thereafter the time for each 3° C rise was noted. Internal temperatures were recorded for the thigh when 95° C in the thigh was the end point temperature and for the *pectoralis major* when 90° C in the breast was the end point. Total, volatile and dripping losses were determined.

Sampling and scoring. An experienced 4-member panel judged the uncarved turkeys for degree of doneness soon after the birds were removed from the oven. Doneness scores were based on a 7-point scale with the optimum represented by the midpoint, 4. The top of the scale, 7, was *very overdone;* the bottom of the scale, 1, *very underdone*.

Turkeys were cooled approximately 2 hr, and the *pectoralis major* and the *gluteus primus* muscles removed for organoleptic tests. For turkeys in Experiment I, judges received slices from the same position of the muscles at each judging period. One half inch cubes were cut from the muscles of the turkeys in Experiments II and III and presented to the judges. A 6-member panel scored the *pectoralis major* samples and a 6-member panel, the *gluteus primus* samples. A 7-point scale with 7 representing the highest value and 1, the lowest was used by the panel to score flavor, juiciness, and tenderness.

[a] Contribution No. 226, Department of Home Economics, Kansas Agricultural Experiment Station, Manhattan.

[b] Presented at the Twentieth Annual Meeting of the Institute of Food Technologists, San Francisco, California, May 16, 1960.

Doneness of meat and doneness of juice were scored similarly to the degree of doneness for uncarved turkeys.

For Experiments II and III, when only 2 turkeys were roasted at each cooking period, at least 1 sample from each treatment was included and either a second or third sample from the other treatment. Four samples always were presented at each organoleptic judging period.

Shear values for 1-in cores from the anterior end of the cooked *pectoralis major* and ½-in cores of the cooked *semitendinosus* muscles were determined on a Warner-Bratzler shearing apparatus. Press fluid yields for a 25-g ground sample of the *pectoralis major* muscle were determined with a Carver laboratory press.

Other procedures were similar to those described by Goertz, Cooley, Ferguson, and Harrison *(2)*.

Statistical analyses. For fresh-unfrozen vs frozen turkeys (Experiments I and II) data obtained within each experiment were subjected to analyses of variance to determine the effect of treatment on: (1) cooking time, 2) cooking losses, (3) shear values, (4) press fluid yields, (5) doneness, and (6) palatability factors. The t-test was used to analyze data for these factors for the turkeys roasted with the breast-up vs the breast-down (Experiment III).

When appropriate, least significant differences were calculated for data for turkeys in Experiment I. Data for turkeys in Experiment II required 3 least significant differences ($P<.05$) for each factor. This was necessary for comparison of treatment groups, because 12 turkeys were used for the fresh-unfrozen group, 12 for fresh-frozen, 8 for 1 mo storage, and 8 for 3 mo at 0° F. The least significant differences for "12, 12" were used to compare data for fresh-unfrozen and fresh-frozen turkeys and for "8, 8", for 1 and 3 mo storage turkeys.

Histological study. Histological samples were taken from the posterior end of the *pectoralis major* and from the lateral side of the proximal end of the *gluteus primus* muscles of turkeys in Experiment II before and after roasting. Samples were fixed in physiological salt and 10% formalin solution. Longitudinal sections were cut on a freezing microtome and stained for muscle tissue and fat with Harris' hemotoxylin and Herxheimers scarlet red, respectively. The stained sections were teased onto slides and mounted in glycerine jelly.

RESULTS AND DISCUSSION

As in previous work with poultry reported from this laboratory *(2)*, the terms *pectoralis major* and *thigh* will be used to denote the muscle in which the end point temperature was taken. The terms *light* and *dark* meat refer to the samples removed from the birds irrespective of the muscle in which the end point of roasting was determined.

Fresh-unfrozen vs fresh-frozen, Experiment I. End point temperatures of 90° C in the *pectoralis major* and of 95° C in the thigh muscles were used for fresh-unfrozen and for fresh-frozen turkeys (Table 1). When doneness of the uncarved turkeys was evaluated by feel of drumstick, appearance of the thigh joint, and movement of the leg joint, turkeys roasted to 95° C in the thigh generally were significantly ($P<.05$) more done than those cooked to 90° C in the *pectoralis major*. This was true for fresh-unfrozen as well as fresh-frozen turkeys. Doneness scores for dark meat samples also were significantly ($P<.05$) higher for turkeys roasted to 95° C in the thigh than for that from birds roasted to 90° C in the *pectoralis major*. Turkeys roasted to 95° C in the thigh were nearer optimum doneness than those cooked to 90° C in the *pectoralis major*.

Total cooking time in min/lb was significantly ($P<.05$) longer for fresh-unfrozen turkeys roasted to 95° C in the thigh than for fresh-unfrozen turkeys cooked to 90° C in the *pectoralis major*. Likewise, total cooking time for fresh-frozen turkeys roasted to 95° C in the thigh was significantly ($P<.05$) longer than for similar birds roasted to 90° C in the *pectoralis major*. For each end point temperature, cook-

TABLE 1

Average cooking time and losses, doneness and palatability scores, press fluid yields, and shear values for turkeys in Experiment I[1]

Factors[2,3]	Lsd[4]	90° C *Pectoralis major*		95° C Thigh	
		Fresh-unfrozen (10)	Fresh-frozen (10)	Fresh-unfrozen (10)	Fresh-frozen (10)
Total cooking time, min/lb	1.0	13.7	14.4	14.9	15.7
Total cooking losses, %		15.0	15.9	16.0	15.5
Volatile cooking losses, %		8.8	9.6	9.6	9.7
Dripping cooking losses, %		5.9	6.0	6.3	5.8
Doneness before carving					
General appearance		3.6	3.9	3.8	4.1
Fork pricking of drumstick		3.8	3.8	4.0	4.1
Feel of drumstick	0.3	3.6	3.7	4.1	4.1
Movement of leg joints	0.4	3.3	3.5	3.9	3.9
Appearance near joints					
Wing		3.5	3.5	3.8	3.8
Thigh	0.3	3.5	3.6	3.9	3.8
Doneness of juice		3.6	3.6	4.0	3.5
Light meat					
Flavor		5.8	6.1	6.0	5.8
Juiciness		4.8	4.4	4.8	4.2
Press fluid yields, ml/25 g		8.3	8.4	8.2	8.5
Tenderness		6.1	6.1	6.2	6.0
Shear value, 1 in core	1.0	8.1	7.7	6.8	7.9
Doneness		3.9	4.1	4.1	3.9
Dark meat					
Flavor		5.8	5.9	5.9	6.0
Juiciness		5.3	5.2	5.1	4.9
Tenderness		5.7	5.7	6.0	5.9
Doneness	0.4	3.5	3.7	4.1	4.1

[1] Numbers in parentheses indicate number of turkeys in each treatment.

[2] Flavor, juiciness, and tenderness scores, range 7–1.

[3] Doneness scores based on a 7-point scale with a midpoint of 4 indicating optimum doneness; smaller number represent underdoneness and larger numbers, overdoneness.

[4] Least significant difference ($P<.05$).

ing times were slightly, but not significantly less for fresh-unfrozen than for fresh-frozen turkeys.

Total, volatile, and dripping cooking losses, press fluid yields (light meat), and flavor scores for both light and dark meat were about the same for fresh-unfrozen and fresh-frozen turkeys roasted to either 90° C in the *pectoralis major* or to 95° C in the thigh. Juiciness scores for light meat were higher for fresh-unfrozen than for those of fresh-frozen turkeys; however, this was not a significant difference.

Shear values for light meat cores from turkeys roasted to 90° C in the *pectoralis major* and for those from the fresh-frozen turkeys roasted to 95° C in the thigh generally were significantly ($P<.05$) higher, indicating lower tenderness, than for those from fresh-unfrozen turkeys roasted to 95° C in the thigh. Conversely, tenderness scores for light meat were similar for all turkeys as were those for dark meat from all turkeys.

Fresh-unfrozen vs frozen, Experiment II. All turkeys in this experiment were roasted to an internal temperature of 95° C in the thigh, because this end point produced birds nearer the concept of optimum doneness than those in Experiment I roasted to 90° C in the *pectoralis major* muscle. Fresh-unfrozen birds again were compared with fresh-frozen turkeys. In addition, a group from the same lot as the fresh-unfrozen and fresh-frozen was tested following 1 and 3 mo storage at 0° F.

The initial temperature of the right thigh for all turkeys in this group was 15° ± 1° C. Curves for the rate of heat penetration (Figure 1) were similar for the fresh-unfrozen and fresh-frozen turkeys, with that of the fresh-frozen group slightly less steep than the curve of the fresh-unfrozen group. The curves for the 1 and 3 mo turkeys were similar and always less steep than those of either the fresh-unfrozen or fresh-frozen turkeys.

Total cooking time in min/lb was significantly ($P<.05$) shorter for fresh-unfrozen turkeys than for any of the frozen defrosted birds (Table 2). Also, the turkeys stored for 3 mo at 0° F required a significantly ($P<.05$) longer roasting time than did fresh-frozen or 1 mo turkeys. Total cooking losses were similar for all turkeys. Volatile losses were lowest and dripping losses highest for fresh-unfrozen turkeys and these generally were significantly ($P<.05$) different from those for the frozen defrosted turkeys.

Doneness scores for light and dark meat and for movement of the thigh joint generally were significantly ($P<.05$) higher for the turkeys frozen 1 mo than for those for any of the other treatment groups. Flavor scores for the light meat were highest for those scored as most done (turkeys stored 1 mo) and were significantly ($P<.05$) higher than those for fresh-unfrozen and fresh-frozen turkeys. Light meat juiciness scores were significantly ($P<.05$) lower for the turkeys stored 3 mo than for those for any of the other turkeys in Experiment II. However, press fluid yields that often are associated with juiciness scores were about the same for all turkeys as were light meat tenderness scores and shear values.

Tenderness scores for dark meat of turkey stored at 0° F for 1 and 3 mo were significantly ($P<.05$) higher than those for the fresh-unfrozen and fresh-frozen turkeys. Shear values for ½-in cores of dark meat were lowest for turkeys stored for 3 mo and significantly ($P<.05$) lower than those from turkeys stored 1 mo at 0° F. Flavor scores for dark meat were higher for fresh-unfrozen than for those of the frozen turkeys; however this difference was not significant.

Histological characteristics of muscle fibers, connective tissue, and fat in the *pectoralis major* and *gluteus primus* muscles were studied. Muscle fibers and fat deposition appeared similar in cooked samples for most of the turkeys in Experiment II. Both granulated and banded forms of collagenous connective tissue were present in samples from the fresh-unfrozen and fresh-frozen turkeys. Only the granulated form of collagenous connective tissue was observed in cooked samples from turkeys stored at 0° F for 1 or 3 mo.

Breast-up vs breast-down, Experiment III. Turkeys were roasted breast-up for the entire roasting period and compared with those cooked breast-down during the first hour of roasting. Birds in the latter

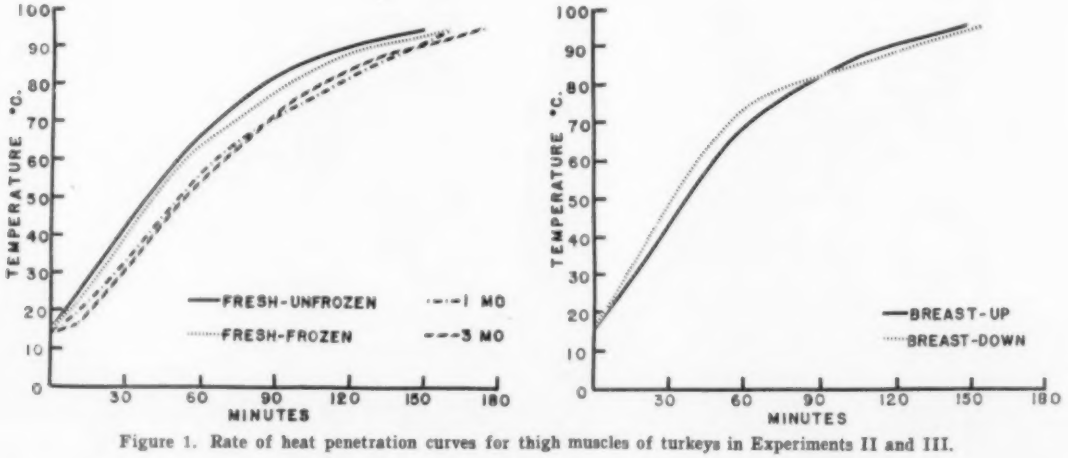

Figure 1. Rate of heat penetration curves for thigh muscles of turkeys in Experiments II and III.

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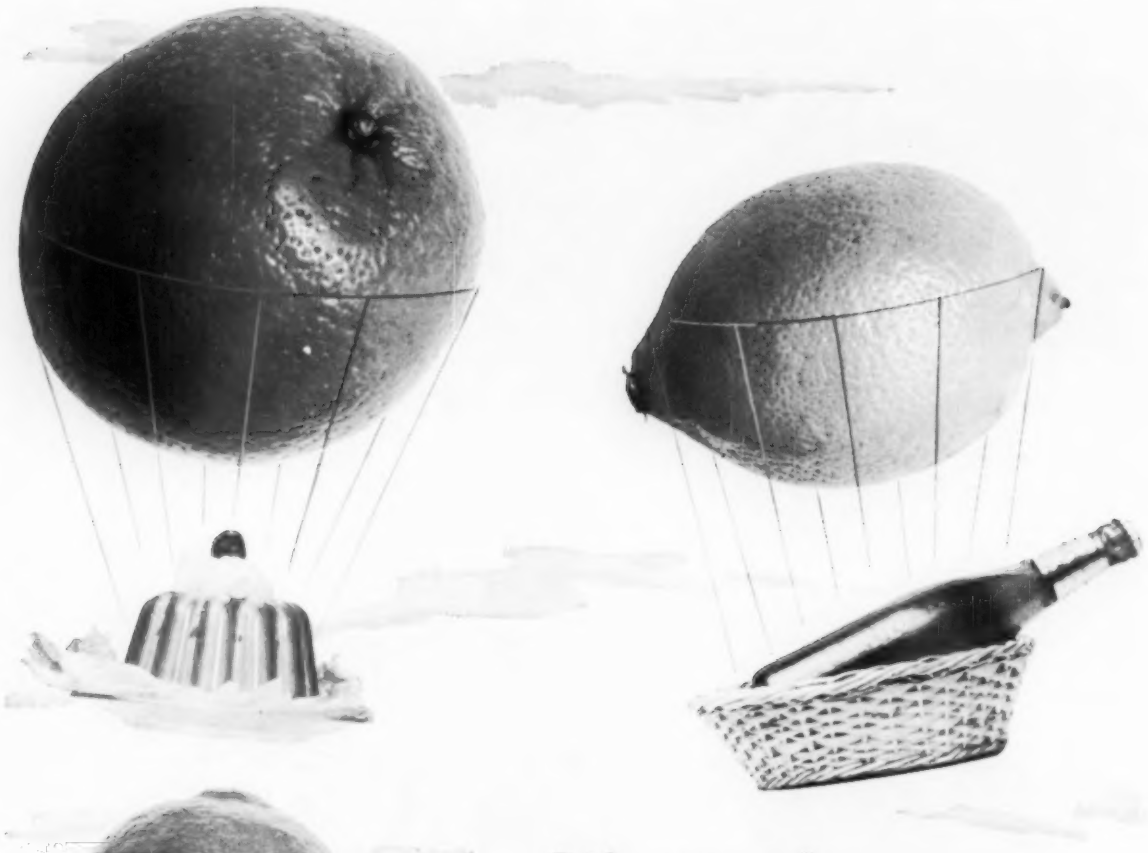

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TABLE 2

Average cooking time and losses, doneness and palatability scores, press fluid yields, and shear values for turkeys in Experiment II[1]

Factors[2,3]	Lsds[4]			Fresh-unfrozen	Frozen		
	(12, 12)	(8, 8)	(8, 12)	(12)	Fresh (12)	1 mo (8)	3 mo (8)
Total cooking time, min/lb	0.8	1.0	0.9	13.8	15.3	15.1	16.2
Total cooking losses, %				18.1	20.7	19.2	19.8
Volatile cooking losses, %	1.2	1.5	1.3	12.5	15.1	14.8	15.5
Dripping cooking losses, %	1.1	1.3	1.2	5.7	5.3	4.0	4.0
Doneness before carving							
General appearance				3.7	4.1	4.1	3.8
Fork pricking of drumstick				3.9	4.1	4.2	4.0
Feel of drumstick				4.0	4.1	4.2	3.9
Movement of leg joints	0.2	0.3	0.3	3.9	3.8	4.1	3.6
Appearance near joints							
Wing				3.9	3.9	3.9	3.9
Thigh				3.7	3.8	3.9	3.6
Doneness of juice				3.7	3.6	3.7	3.9
Light meat							
Flavor	0.3	0.3	0.3	5.7	5.6	6.1	6.0
Juiciness	0.4	0.5	0.4	4.6	4.7	4.5	4.0
Press fluid yields, ml/25 g				7.6	7.9	7.6	6.9
Tenderness				6.4	6.3	6.2	6.5
Shear value, 1 in core				7.4	8.6	9.1	7.3
Doneness	0.3	0.4	0.3	4.6	4.5	6.2	4.5
Dark meat							
Flavor				6.0	5.3	5.4	5.2
Juiciness				4.3	4.6	4.7	4.3
Tenderness	0.4	0.5	0.4	5.3	5.4	5.8	6.1
Shear value, ½ in core	0.7	0.9	0.8	8.8	8.7	9.4	8.1
Doneness	0.3	0.4	0.3	4.2	4.1	5.8	4.8

[1] Numbers in parentheses indicate the number of turkeys in each treatment.
[2] Flavor, juiciness, and tenderness scores, range 7–1.
[3] Doneness scores based on a 7-point scale with a midpoint of 4 indicating optimum doneness; smaller numbers represent underdoneness and larger numbers, overdoneness.
[4] Least significant difference ($P<.05$).

group were turned after 60 min and cooked breast-up until an end point temperature of 95° C in the thigh was reached.

Average initial temperature in the right thigh was 16° C for turkeys in both groups (breast-up and breast-down) (Figure 1). During the first 80 min of roasting, internal temperatures of the right thigh of the turkeys roasted breast-down were slightly lower than for those roasted breast-up. Thereafter, the internal temperatures of the thighs of both groups were similar.

Total cooking time in min/lb was significantly ($P<.05$) longer for turkeys roasted breast-down than for those cooked breast-up during the entire roasting period (Table 3). It often is assumed that roasting poultry breast-down keeps the light meat from becoming too dry. However, in this study, turkeys roasted breast-down had significantly ($P<.05$) lower juiciness scores for light meat than those roasted breast-up. Doneness scores for unearved turkeys, for light and dark meat, and for juice were similar for turkeys roasted breast-up or breast-down.

Light meat flavor and tenderness scores; dark meat flavor, juiciness, and tenderness scores; press fluid yields; and shear values for light and dark meat were similar for turkeys roasted breast-up or breast-down. Lowe *(3)* also reported no significant differences in flavor, juiciness, or tenderness when working with roasters cooked breast-up or breast-down. However, she noted that shear values, press fluid yields, and palatability scores were slightly in favor of roasters cooked breast-down. In the study reported here, press fluid yields for the light meat of turkeys were slightly higher for those roasted breast-down than for those cooked breast-up. Shear values for 1 in and ½-in

TABLE 3

Average cooking time and losses, doneness and palatability scores, press fluid yields, and shear values for turkeys in Experiment III[1]

Factors[2,3]	Breast-up (9)	Breast-down (9)
Total cooking time, min/lb	14.9	16.7 *
Total cooking losses, %	19.4	20.2
Volatile cooking losses, %	14.2	16.0
Dripping cooking losses, %	4.8	3.8
Doneness before carving		
General appearance	3.6	3.8
Fork pricking of drumstick	3.9	3.7
Feel of drumstick	3.9	3.8
Movement of leg joints	3.8	3.8
Appearance near joints		
Wing	3.5	3.5
Thigh	3.7	3.8
Back	3.2	3.4
Doneness of juice	3.8	3.8
Light meat		
Flavor	5.8	5.7
Juiciness	4.9	4.4 *
Press fluid yields, ml/25 g	7.3	7.6
Tenderness	6.0	6.0
Shear value, 1 in core	11.3	12.4
Doneness	4.2	4.1
Dark meat		
Flavor	5.4	5.6
Juiciness	4.7	5.0
Tenderness	6.0	6.1
Shear value, ½ in core	6.8	7.9
Doneness	4.5	4.3

[1] Numbers in parentheses indicate the number of turkeys in each treatment.
[2] Flavor, juiciness, and tenderness scores, range 7–1.
[3] Doneness scores based on a 7-point scale with a midpoint of 4 indicating optimum doneness; smaller numbers represent underdoneness and larger numbers, overdoneness.
* ($P<.05$).

cores from the light and dark meat, respectively, also were higher (indicating less tender meat) for the turkeys roasted breast-down than for those roasted breast-up.

SUMMARY

Fresh-unfrozen and fresh-frozen turkeys were roasted to 90° C in the breast and to 95° C in the thigh. Turkeys roasted to 95° C in the thigh generally were more done than thoes roasted to 90° C in the breast. Total cooking time was longer ($P<.05$) for fresh-frozen turkeys roasted to 95° C in the thigh than for fresh-frozen turkeys roasted to 90° C in the breast. Similar results for cooking time were noted for fresh-unfrozen turkeys.

Also, fresh-unfrozen and fresh-frozen turkeys and turkeys stored 1 and 3 months at 0° F were roasted to 95° C in the thigh. Total cooking time was shorter ($P<.05$) and volatile cooking losses lower ($P<.05$) for fresh-unfrozen turkeys than for the frozen turkeys. Dripping losses were lower ($P<.05$) and light meat flavor scores and dark meat tenderness scores higher ($P<.05$) for turkeys stored 1 and 3 months than for the fresh-unforzen or fresh-frozen turkeys. Doneness scores for light and dark meat were higher ($P<.05$) for turkeys stored 1 month than for those of the other turkeys; whereas doneness scores for uncarved turkeys generally were similar.

In addition, frozen, defrosted turkeys were roasted breast-up or breast-down. The thigh temperature of turkeys roasted breast-up increased more rapidly and cooking time was shorter ($P<.05$) for turkeys roasted breast-up than for those roasted breast-down. Doneness and other palatability scores generally were similar for all birds in this group.

Acknowledgment

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The Relationship of Apple Maturity to Applesauce Quality[a, b]

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THE PREDICTION and evaluation of apple maturity at harvest are matters of considerable commercial importance. In New York State alone seven million bushels of apples are processed, about half of these into sauce. Selection of harvest date, as well as subsequent storage conditions and duration prior to processing for this fruit, continues to be a vexing and incompletely understood problem.

To be sure, many factors must be considered in such decisions. The fruit-grower may understand that larger apples and hence greater yield can be obtained by delaying the harvest, but at the same time he may anticipate excessive drop or wish to take advantage of a favorable early-season market. The availability of harvest labor at a certain time may be of overriding importance to him. It is further possible that the processor may be interested primarily in insuring early procurement of his own needs or in securing apples immature enough to be very firm or to afford a long storage life. Again storage time and conditions may be more a matter of economy, plant capacity, or facilities available for storage and handling than of scheduling apples for processing at optimum maturity. Nevertheless, the effect of maturity on the quality of the final product has been of increasing concern—a concern in which the enlightened consumer has a high stake.

The study reported here deals with the effect of apple maturity on sauce quality. It is concerned as well with indices which may be used to evaluate and predict maturity in the fresh apple so as to achieve the highest quality in the processed product.

This problem is not a new one in the research annals of horticulture and food technology. Concern over climatological effects on plant growth, if not maturity, was expressed in the literature early in the century, notably by Livingston *(5)*. This interest was extended to the ripening of fruit by Tufts *(11)* in 1930. Then Winkler *(14)* in 1932 introduced Balling-acid ratio as an index of maturity for Tokay grapes. Later, with Williams *(15)*, he related this ratio to heat unit accumulation in the 28 days preceding harvest. Magness and Taylor *(7)* in 1925 recommended the use of the now-popular pressure tester for the determination of fruit maturity, and Haller *(3)* later extended its application to storage apples. Magness *et al (6)* in 1926 recognized the increase in sugar content and the decrease in titratable acidity which accompany the ripening of apples in storage.

In 1942 Tukey *(12)* suggested that days from full bloom was a better indication of maturity than calendar date. Smock *(8)* in 1948 published a comprehensive study of maturity indices for determining the optimum picking date for McIntosh apples. These included pressure test, ease of separation when picking, ground color, soluble solids, days from full bloom, calendar date, and respiration rate. He concluded that respiration rate and ground color were the best indices. However, this study was only of apples for the fresh market, rather than for processing. Smock and Neubert *(9)* later point out that soluble solids content has limited value as a maturity index because of variations from year to year.

As the processing of apples has become more important commercially, the emphasis has switched to the effect of maturity on quality of the processed product. Caldwell *et al (1)* reported in 1955 on the relationship of frozen slice quality to ripeness and concluded that ripening of firm-ripe fruit was required to improve product flavor and, to a lesser extent, color. Wiley and Thompson *(13)* extended the study of maturity and storage effects to canned slices and found that early harvest apples should be ripened 20 days in common storage or 30–60 days in cold storage. However, they found that late harvest apples gave the best product if processed immediately. They suggested that canned-slice quality could be predicted from the raw stock by consideration of sugar and acid content, firmness, color and other factors, perhaps in nomographic form.

EXPERIMENTAL

Two major processing varieties—Rhode Island Greening and Baldwin—were selected for the first part of the maturity study, with the intent of extending the investigation to others later on. The procedure used in 1958 will be described since that used in 1957 differs only in detail. Typical commercial orchards of fully-matured trees near Sodus, New York were selected as sources of fruit. In 1958 the orchards were divided into 2 blocks and in each block a group of 3 trees was selected for each intended harvest date. Apples from each block retained their identity throughout storage and processing and hence provided replication in the experiment.

At each harvest 9 bushels of fruit were picked, promptly delivered to the Experiment Station, and machine size-graded. Two-bushel lots made up from the prevailing proportions of the 3 dominant sizes were set aside for processing the following day, for outdoor storage, and for cold storage. Samples of 25 apples were drawn for pressure test determination and for laboratory analysis. The analysis of the fresh apples consisted of the following:

Total acid as malic by titration with NaOH and phenolphthalein.

Soluble solids by refractometer.

Flesh color by Hunter Color Difference Meter.

[a] Presented at the Twentieth Annual Meeting of the Institute of Food Technologists, San Francisco, May 17, 1960.

[b] Approved by the director of the New York State Agricultural Experiment Station as Journal Paper No. 1229, July 14, 1960.

The apples in 25-kg. lots were processed into sauce in a pilot plant of 300-lb per hour capacity. After peeling and coring in a Pease machine, the apples were moderately hand-trimmed, rinsed, and elevated into an Urschel Model B cutter adjusted to give slices $^{3}/_{16}$ x $1^{1}/_{4}$ in x random length. These slices discharged from the cutter directly into a continuous screw cooker where they were cooked by heating to 213° F in a 3-min passage through the cooker. As in the familiar Allen cooker, heating is accomplished by injecting steam directly into the slices. In this instance steam entered through the perforated false bottom of the 4-ft long by 4-in diameter cooker trough while the slices were subjected to the slow mixing and conveying action of a ribbon screw. The cooked slices discharged directly into a Langsenkamp laboratory finisher operated at 1000 rpm with a $^{3}/_{32}$-in screen. Such high-speed operation of the finisher was required to obtain the peripheral speed and the proportion of waste common to commercial operation in finishers of larger diameter.

The sauce discharged from the finisher at 180° F, was weighed, and was subsequently batched in a jacketed kettle. There it was held in the range 160°–170° F while the consistency and soluble solids were carefully adjusted by the addition of calculated amounts of warm water and granulated cane sugar. Consistency was adjusted to a Bostwick of 4.6 (10 sec at 150° F), resulting in a final reading of 3.4 at 30° C. The per cent soluble solids was adjusted to about 19.

Following these adjustments the sauce was pumped with a Moyno pump through a shell-and-tube heat exchanger consisting of 10 ft of steam-heated length of $^{5}/_{8}$ in O.D. stainless steel tubing. The sauce, reheated to 195° F, was then filled directly into 307 x 409 cans, which were subsequently closed, rolled on a holding belt for 3 min, and cooled in a tank of air-agitated water.

The batch-wise adjustment of consistency and solids was resorted to because of the difficulty in establishing controlled proportioning of sugar and water in a minor fraction of the time required to pass the 25-kg experimental lot through the cooker,—about 15 min. Reheating of the product before filling, another departure from commercial practice, is necessary to reach the required filling temperature. The ratio of product mass to equipment mass is much lower in pilot plant operation and results in excessive heat loss by the product.

Presure tests were obtained on 25-apple samples by taking 4 readings per apple with the Magness and Taylor-type 30-lb tester equipped with the $^{7}/_{16}$-in head. Samples were measured periodically during storage in 1958 to follow closely the ripening of the fruit. The lots being held in outdoor and in cold storage were processed when extrapolation of this pressure test data indicated 15 lb would be reached.

The sauce was analyzed in the laboratory after a few months storage to determine, as in the fresh apples, total acid and soluble solids. Sauce color was read on the Hunter Color Difference Meter and consistency was measured with the Bostwick consistometer. In order to determine objectively the grain of the sauce, the sieve method of Kimball and Kertesz was employed. Particle size is expressed as the diameter in millimeters which divides all larger and smaller particles into equal volumes.

Subjective analysis of the sauce was provided by submitting one can of each lot to processed product inspectors. The sauce was scored by them according to the "U.S. Standards for Grades of Canned Applesauce."

Heat units or degree days were calculated as the difference between the base of 40° F and the mean of the daily maximum and minimum temperatures. They were accumulated from April 1 of each year till the day of harvest or of processing. In order to account for ripening in storage at temperatures below 40° F, an alternate base of 26° F was used when calculating heat units for apples held in storage in 1958.

RESULTS

Pressure test. At the very outset this study required an effective means of expressing the maturity of the fruit from the processing standpoint. This would serve as the pivotal factor against which both an index of seasonal effectiveness in promoting maturation and the ultimate quality of the processed product could be compared. Although pressure test was originally selected for this role, it was later apparent that this particular index did not in fact correlate well with fruit maturity at harvest.

In Figure 1 a family of pressure test curves are shown for a series of harvests of R. I. Greenings made in 1958. It is clear

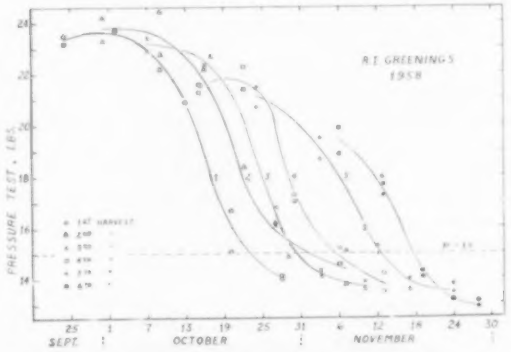

Figure 1. Pressure test relationships for duplicate lots of R. I. Greenings harvested at intervals in 1958 and held in outdoor storage.

that the change in pressure test during the first few harvests was so slight as to be illusive and easily masked, e.g. by sampling error. This period, characterized by a low rate of change of pressure test, unfortunately occurs just prior to and at the usual harvest time when an effective index of maturity is especially required for prediction and selection of harvest date. It was only after these early harvest apples had been in storage a week or two that ripening proceeded at a much faster rate, thence tending to level out again after the pressure test had dropped to the 14-lb level.

Soluble solids-acid ratio. This negative result forced us in retrospect to look for a more effective index of maturity at harvest time. Such chemical indices as sugar or acid content of the fruit suggested themselves because of their direct bearing on the edible quality of the product, both fresh and processed. Unfortunately the ranges, in which they vary during the season, themselves change from season to season with fruit size or photosynthetic activity. However, this shortcoming can be corrected by forming the ratio of soluble solids with total acid. Soluble solids was selected here because it is more readily measured than is sugar. This soluble solids-acid ratio is then, more constant from season to season than the absolute level of either component alone. Furthermore, soluble solids is increasing as total acid is decreasing with maturation, so that the ratio itself is changing more rapidly than either. For these reasons soluble solids-acid ratio replaced pressure test in this study as the primary index of fruit maturity.

As a first check on the effectiveness of this index, the ratios obtained for a series of harvests of both R. I. Greenings and Baldwins in 1957 and 1958 were plotted against the calendar date of harvest in Figure 2. While the ratios gave very decided trends for each variety in each year, the relationships for neither varieties nor years coincided. There was a displacement of about 2 weeks between the curves for 1957 and 1958 in each variety. It was noted that 1958 had been a near-record cool summer while 1957 had been about normal with respect to temperature. Heat unit accumulation had in fact lagged in 1958 about two weeks behind 1957. Consequently, when soluble solids-acid ratios are plotted against heat unit accumulation in Figure 3, the trends for the 2 years coincide, describing but two curves—one for each variety. The corresponding linear coefficients of correlation are 0.91 for R. I. Greenings and 0.95 for Baldwins.

Sauce color and flavor. Since we have tentatively established heat units as a seasonal index of apple maturity, better than the calendar date of harvest, we may proceed to show how the

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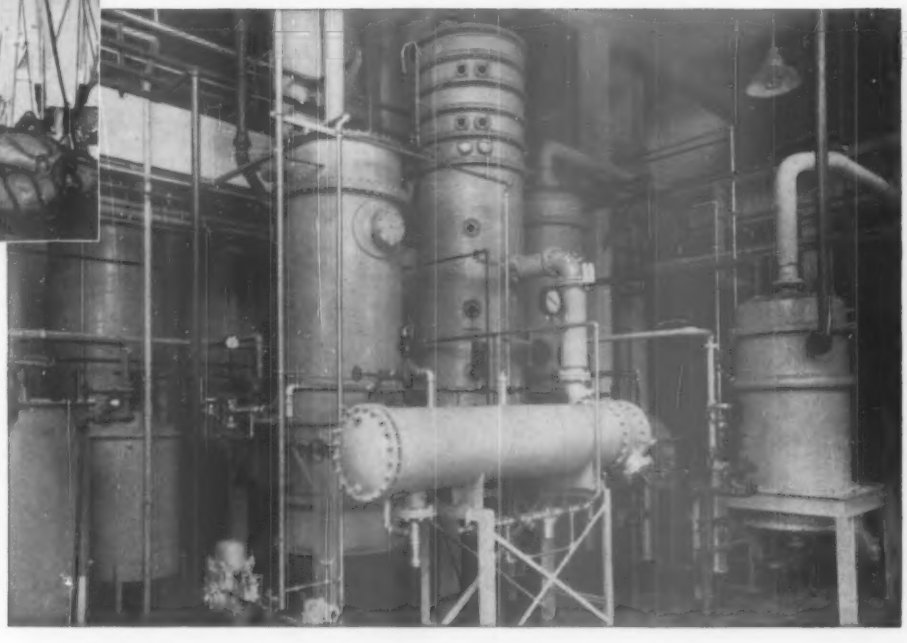

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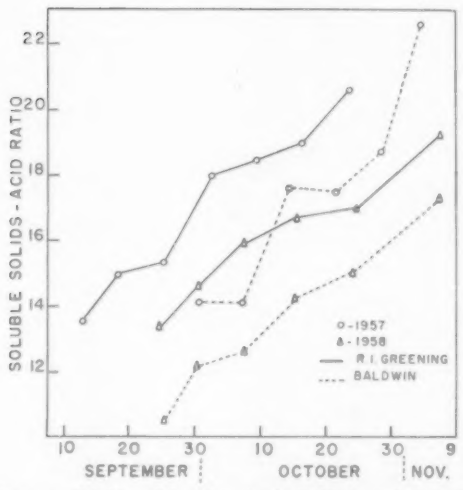

Figure 2. Soluble solid-acid ratios in Baldwin and R. I. Greening apples for a series of harvests in 1957 and 1958.

color and flavor of the processed product are related to maturity expressed in terms of heat units. Color and flavor scores obtained under the processed product grade for sauce made the day following harvest are plotted against accumulated heat units in Figure 4. In this sauce made from R. I. Greenings both factors were initially substandard; but the quality improved with maturation so that by the time 3800 heat units had accumulated in the orchard in 1958 a Grade A sauce was made without the necessity for ripening the apples in storage. For Baldwin apples the corresponding cross-over occurred in 1958 at 4000 heat units.

Since most processing apples are storage-ripened, it is pertinent to inquire how this post-harvest ripening affects sauce quality. When apples from the same harvests were ripened in outdoor or in cold storage to a pressure test of 15 lb, the improvement in sauce quality with maturity of harvest persisted but was less pronounced as shown in Table 1. Now only the first harvest fails to provide a Grade A product.

Note that heat unit totals for the storage apples are approximately level for each variety, averaging 4,360 for R. I. Greenings and 4,660 for Baldwins. As expected, the apples

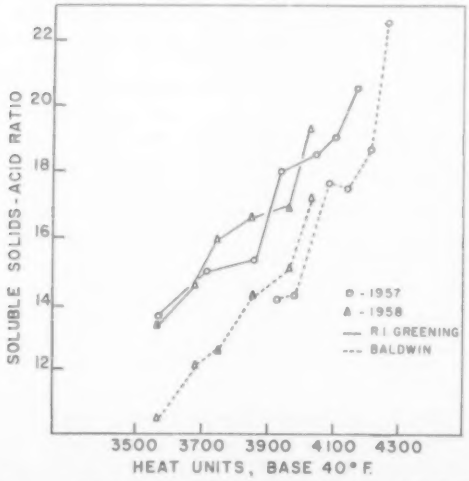

Figure 3. Soluble solid-acid ratios in Baldwin and R. I. Greening apples as a function of accumulated heat units.

TABLE 1

Average color and flavor scores for apples ripened to 15 pounds pressure test in both outdoor and cold storages in 1958

Harvest date	R. I. Greening			Baldwin		
	Heat units[1]	Color	Flavor	Heat units[1]	Color	Flavor
9/24	4,344	16.8	17.0	4,674	16.5	16.8
9/30	4,468	17.2	17.0	4,750	17.2	17.3
10/7	4,328	17.0	17.2	4,683	17.5	17.8
10/15	4,324	18.0	18.0	4,597	17.8	18.0
10/24	4,363	18.8	18.0	4,608	18.5	17.8
11/5	4,331	19.0	18.0	4,650	18.5	18.2
Averages	4,360			4,660		

[1] Base 40° F in orchard; base 26° F in storage. Data are averages of two storages and two replicates.

ripened longest on the tree required least ripening in storage. Even though different heat unit bases, 40° F and 26° F, were used for orchard and storage, the relative constancy of these heat unit totals within each variety shows the complementary character of orchard and storage ripening. The second harvest apples, which were a significant exception, ripened most slowly and provided the longest storage life.

Comparison of maturity indices. Both soluble solids-acid ratio and heat units have been suggested as indices of maturity for processing apples in this study. It has been shown that if the former is a measure of the chemical maturity of the fruit, then heat unit accumulation, which correlated closely with it in this 2-year study, is a better-seasonal measure of maturity than is calendar date.

Another index of maturity is days following full bloom, which like heat unit accumulation is a seasonal measure. This index has been employed by others and deserves consideration. In Table 2 these 3 indices together with pressure test are listed for comparison.

TABLE 2

Comparison of maturity indices from 1957 and 1958 data on R. I. Greenings

	1957	1958	1957	1958	1957
Heat units (base 40° F)	3935	3800	3800	3555	3975
Days after full bloom	140	140	131	122	142
Calendar date of harvest	10/2	10/11	9/23	9/23	10/4
Pressure test, lb	24.3	22.1	25.1	24.1	24.1
Soluble solids/acid	17.1	16.3	15.4	13.5	17.4
Apple flesh color, Hunter a/b	−.25	−.29	−.32	−.38	−.23

Supplementing soluble solids-acid ratio here as a direct measure of fruit maturity and quality is the hue of apple flesh color, or a/b ratio, as determined with the Hunter Color Difference Meter. This objective measure has been observed to undergo a decided change from green-yellow to yellow as apples ripen. However, it may be more of a research tool than a practical index of maturity.

Adjacent columns in the table contain data sets from both seasons selected so that the underlined index remains constant. This permits ready inspection of how closely the other indices agree in the 2 seasons. It is at once apparent that no 2 indices agree exactly. The greatest differences in soluble solids-acid ratio and flesh color, both sensitive measures of fruit maturity or quality, occur when calendar date of harvest or pressure test are held constant. It has already been shown that these 2 indices are inadequate.

There is little to choose in this respect between heat units and date of full bloom. It is noteworthy that the differences observed in soluble solids-acid ratio and in flesh color occur to equal extent but in opposite directions. At equal heat unit accumulation apples were less ripe in 1957 than in 1958. On the contrary at an equal number of days after bloom, apples were more ripe in 1957 than in 1958. By employing in 1958 a compromise between the 2 indices, heat units and days after full bloom—say 3870 and 145 respectively, R. I. Greenings with

the same flavor and color as in the previous year could have been harvested. This suggests that the truth lies somewhere in between these 2 indices.

In the preceding comparison we have relied upon soluble solids-acid ratio and the hue of flesh color as measures of raw product quality. This comparison is strengthened by referring further to the quality of the processed product—applesauce. This can be done indirectly by noting that these measurements of raw product quality correlate with flavor and color scores in the sauce with coefficients of 0.87 and 0.80 respectively.

Other measurements of sauce quality. Relating apple maturity to sauce quality implies adequate measurement of the latter. However, the several quality factors differed both in their susceptibility to adequate objective measurement and in their sensitivity to the necessarily combined effects of maturity and processing.

Consistency of the sauce was successfully controlled at a certain level and hence was eliminated in this study as a factor in which maturity affects quality. In 72 lots of sauce made from R. I. Greenings and Baldwins in 1958, the change in Bostwick consistency reading was related to the addition of water with a linear coefficient of 0.91. The measures taken to control consistency—adding more or less water—necessarily affected sauce yield, and this will be separately reported.

Although the measurements of particle size, grain, or finish in these sauces do not show large differences due to maturity, both the objective determination of particle size and scores for finish given by the processed product inspectors show a slight but consistent trend towards improvement in grain with advancing maturity as shown in Table 3. This trend is most

TABLE 3

Effect of harvest sequence on the score for finish in applesauce when processed on the day following harvest

Harvest	R. I. Greening				Baldwin			
	Particle size, mm		Finish score		Particle size, mm		Finish score	
1957								
1	1.00		15		1.04		17	
2	1.07	1.03	16	16.0	1.10	1.09	17	17.0
3	1.02		17		1.13		17	
4	1.05		18		1.17		17	
5	1.12	1.07	18	18.0	1.14	1.14	18	17.7
6	1.04		18		1.12		18	
1958								
1	1.03		18		.75		17	
2	1.02	1.00	17½	17.7	.74	.78	16½	16.8
3	.94		17½		.84		17	
4	1.11		18		1.16		17½	
5	1.10	1.09	18	18.2	.86	.99	17½	17.7
6	1.07		18½		.94		18	

Data are averages of duplicates.

clearly shown if the averages of the first 3 and last 3 harvests are compared in each case. It is then clear that the improvement is consistent in all 8 comparisons. The average increase in particle size is 0.10, or 10%, and the average improvement in the grade score for finish is 1.0, sufficient to advance the grade from C to A.

This improvement is seen only in sauce processed directly after harvest. When these apples were ripened in storage to 15-lb pressure test, the finish was substantially the same in sauces from all harvests. Moreover, there does not seem in these data to be any certain level of particle size associated with a Grade A product. The objective test fails to differentiate absolutely between a high and a low grade finish.

Color and flavor in sauce proved to be much more easily or reliably judged by eye and tongue than by objective measurements. Hence reference is made in this respect to processed product grade scores when evaluating the effects of maturity on sauce quality. Measurements made with the Hunter Color Difference Meter on sauce failed to differentiate in any of the 3 parameters—hue, chroma, or brightness—among color differences which to the eye, even in color photographs, were readily apparent. Only in the fresh apple was the difference in hue detected with the Hunter.

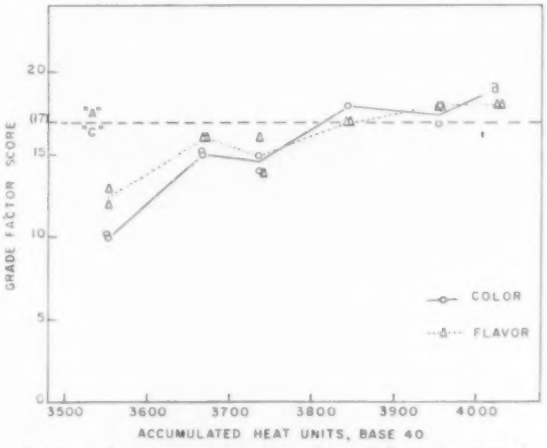

Figure 4. Improvement in sauce color and flavor in successive harvests of R. I. Greenings, 1958.

DISCUSSION

The inability of the pressure test to indicate harvest maturity effectively came as no great surprise. Previous experience with its uncertainty at harvest time and considerable distrust of it among growers and processors had been attributed to inadequate sampling. However, in this case sampling was carried out with great care, replication was provided, and tests were repeated frequently throughout the harvest and storage periods. The well-defined curves provided by this considerable body of pressure test data clearly indicated the source of difficulty—the initial low rate of change. Thus, it seems fair to say that pressure test is a better index of maturity for apples ripening in storage than for apples approaching harvest maturity on the tree.

Soluble solids-acid ratio, though requiring only a refractometer reading and a simple acid-base titration, is less simple than pressure test to perform in the field. It is a method for the processor, rather than the grower, to use in following and predicting maturation or in evaluating the potential quality in a block of apples acquired for processing. However, we have demonstrated a close relationship between soluble solids-acid ratio and heat unit accumulation. The latter is easily followed from day to day by either processor or grower and may be particularly useful for predicting the time of maturation, as well as for present evaluation. Of course, we must make some reservation here for the abnormal orchard in which maturation is affected one way or the other by disease or by some cultural aberration such as excessive fertilization.

In the light of results subsequently obtained in the abnormally-warm 1959 season, further reservation on the use of heat unit accumulation as a maturity index may be necessary. It appears that the conventional use of a linear heat unit function may not be applica-

ble when high temperatures prevail. This development will be reported separately.

Pressure test and soluble solids-acid ratio have separate applications in quality control when proper processing maturity has been reached. From the pressure test, the duration and temperature of the cook necessary to achieve the desired texture or grain of the product can be deduced. On the other hand the prevailing soluble solids-acid ratio will inform the processor of the proportion of sugar required to reach a certain level of soluble solids, or preferably a certain ratio in the product. Fortunately the acceptable range is quite broad, with a peak being variously reported as high as 49 *(2)* and as low as 25.5–32.8 *(10)*. In data collected here from a small expert taste panel rather than from a large, consumer panel, greatest acceptance was obtained for soluble solids-acid ratios in the range from 28 to 45 as seen in Figure 5.

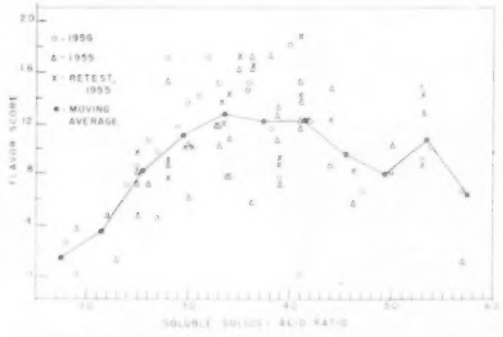

Figure 5. Comparison of taste panel flavor score with soluble solids-acid ratio in applesauce.

It is important to note that while wide differences were encountered in sauce quality when apples were processed the day following harvest, storage ripening the fruit to a common pressure test or heat unit accumulation prior to processing greatly reduced these differences. Even so, storage-ripened apples never developed the desirable color, flavor, and grain encountered in tree-ripened fruit. This discrepancy was readily apparent in the sauce. Clearly from the standpoint of product quality, R. I. Greenings should be tree-ripened further than is now current. Ordinarily this variety is harvested late in September in the Lake Ontario fruit belt. Our results suggest that this harvest should be delayed a week or two, depending on heat-unit accumulation, in order to maximize product quality. Since the fruit is still growing at this time, this might also increase orchard yield, barring excessive drops.

The possibility of assessing maturation of apples in storage from further heat unit accumulation is especially interesting. Where storage temperature is closely controlled, as in cold storage, this would make it possible to predict accurately the time at which storage apples would reach the optimum ripeness for processing based on previous experience. A stumbling block here is the selection of the proper base temperature for calculating heat units in storage. In this study 26° F was arbitrarily selected since it was below the lowest storage temperature and approximated the freezing point where a discontinuity in ripening could be expected. It also was seen to provide nearly equal heat unit accumulations in outdoor and cold storage. Appropriately, ripening rates were about three times as fast in outdoor storage as in cold storage, with average temperatures of 48° F and 33° F, respectively.

SUMMARY

A series of harvests of R. I. Greenings and Baldwin apples in 1957 and 1958 permitted evaluation of maturity indices. Their relationship to the quality of sauce processed from these apples both with and without storage ripening was also investigated. An applesauce pilot plant of 300 lb per hour capacity was used to process sauce from this fruit under precisely controlled conditions and in equipment closely simulating commercial processing.

Pressure test was discarded as a prime index of maturity at harvest in favor of soluble solids-acid ratio in the fresh fruit. It was shown that the rate of change of pressure test prior to and at harvest was too low to provide reliable differences. Conversely, soluble solids-acid ratio changed rapidly in this same period and had a direct bearing on product quality. In addition the ratio was closely correlated with heat unit accumulation over the two seasons studied.

Sauce color, flavor and grain improved as harvest was delayed to allow the fruit to tree-ripen. This was particularly noticeable if the apples were processed into sauce directly after harvest. However, even when the fruit was ripened to a common extent, as measured by pressure test or accumulated heat units, sauce from late-harvest apples was still noticeably better.

While pressure test and calendar date of harvest were shown to be inferior indices of maturity, heat unit accumulation and days after full bloom were observed to be about equally effective. The best prediction of apple maturity in 1958 based on results obtained in 1957 would have involved a compromise between the latter two indices.

Acknowledgments

The authors wish to express their appreciation for the assistance of Floyd P. Foley in the Processed Products Inspection Office of the United States Department of Agriculture at Rochester in inspecting the applesauce. In addition, J. E. Whitcombe; V. Jensen, H. C. Reitmann and P. Nelson were instrumental in carrying out the details of the experiment or in constructing the necessary equipment.

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Optical Density of Tomato Serum from Concentrates as a Measure of Heat Induced Changes in Product Corrosivity*

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(Manuscript received May 18, 1960)

Highly concentrated tomato paste can be stored and shipped more economically than low solids paste. This factor plus recent engineering improvements to concentrating equipment have started a trend toward production of higher solids tomato paste.

Unfortunately, problems of product stability have been encountered with the more concentrated products. Keeping qualities are reduced. Flavor retention of high density pastes becomes critically dependent upon storage temperature. Shelf life of the container is shortened by the evolution of carbon dioxide from the paste, and hydrogen from corrosion of the container.

In order to cope with this problem, packers have found that they must take precautions to see that the paste is not exposed to too much heat during processing, cooling, or storage. This paper is concerned with a method for detecting heat induced changes, and with a basis for quantitatively evaluating these changes.

Luh *et al (1)* have previously reported work on the problem of heat induced changes in tomato paste. They used the changes in amounts of hydroxy methyl furfural, tannin-like compounds, ascorbic acid, amino acids, and serum color to follow the thermal degradation of the paste. Only the serum color is applicable to control uses, for the following reasons:

a. The increase in hydroxy methyl furfural is evidenced only when the storage temperature is above that which might reasonably be encountered.

b. The changes in amino nitrogen, ascorbic acid, polyphenolics and reflectance measurements are all excellent methods for following laboratory type experiments, because the initial values can be determined. In commercial practice, the variation in starting materials makes the use of these methods difficult.

The use of headspace carbon dioxide determination for measuring heat damage also is not feasible, because the amount of carbon dioxide is dependent upon the manner in which the paste was damaged. Therefore, it does not show a linear relationship to degree of darkening change.

The color of tomato serum appears to be the best

* Presented at the Twentieth Annual Meeting of the Institute of Food Technologists, May 18, 1960, San Francisco, California.

current method for commercially evaluating burning of tomato paste. This is because (a) the color of unburned tomato serum after dilution to a given concentration is relatively constant, (b) the amount of change in optical density is proportional to the amount of burning, and (c) the color of tomato serum can be used to make accurate predictions of expected service life.

EXPERIMENTAL METHOD AND RESULTS

The experiments and data described here were designed to clarify two questions:

a. How do solids, per se, affect the shelf life of tomato paste?

b. How do different temperatures during processing, cooling, and storage affect shelf life and product quality?

Terminology as used here:

a. *Optical Density:* 2 minus the log % transmission.

b. *Tomato Serum:* A clear filtrate obtained from tomato juice, puree, or paste, after dilution to 2.5% solids.

c. *Tomato Solids:* The solids of tomatoes as determined by the use of a refractometer and reference to the National Canners Association handbook on tomato products *(2)*.

d. *Normal Paste:* A paste containing 15–30 mg ascorbic acid per 100 g 5% solids, having a U.S.D.A. grade of 57 or 58, and whose serum has an optical density of 0.03 or less after subtracting the blank.

e. *Failed Can:* A can whose end has become distended due to the formation of CO_2 or H_2 inside the container.

Analytical procedures. Optical density of tomato paste serum was determined as follows. After determining the refractometer solids of the paste, 25 g of the paste were diluted with [(10 times % of solids) − 25] ml of H_2O. The diluted juice was then mixed with an equal volume of Celite, and filtered through Whatman No. 42 paper. The OD of the crystal clear filtrate was determined on a Beckman Model B spectrophotometer at 420 mmc in a 1 cm cuvette, using water as a blank. All values reported here have had 0.17 subtracted from them. This was done to correct for the color of normal raw tomato serum.

The choice of 420 mmc as the proper wave length was predicted on the absorption curves shown in Figure 1. This wave length is a compromise between the point of highest absorption at 410 mmc and the lower deviation in the color of raw tomato serum at 425 mmc. Further substantiation for this choice is seen in Table 1, in which the OD of tomato serums from raw tomatoes of a wide variety of types, growing conditions, maturities, and picking dates are reported. These data were also used in determining the amount of correction factor (0.17 OD)

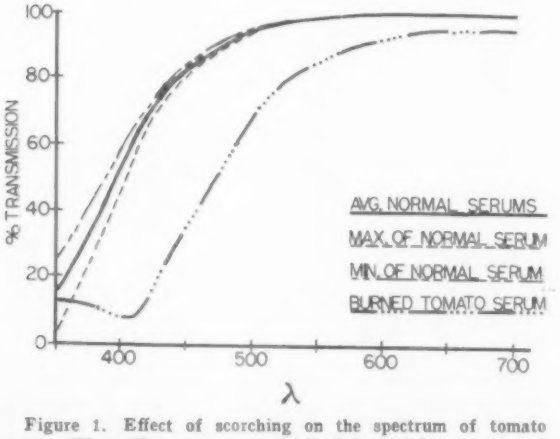

Figure 1. Effect of scorching on the spectrum of tomato serum. "Normal" serums were obtained from juice, puree, and paste serums. "Burned" paste was obtained by storing a 36% paste for 10 days at 130° F.

TABLE 1

Optical density at 420 millimicrons in 1.0 cm cuvette of raw tomato serum

Tomato type	Size (Inches)	Growing area (California)	Picking date	Refractometer solids	Optical density[1] of 2.5% total solids samples
Green Pearson Improved	2	Tracy	10/2/55	4.4	0.17
Pink Pearson Improved	2	Tracy	10/2/55	5.7	0.17
Ripe Pearson Improved	2	Tracy	10/2/55	4.8	0.15
Ripe Pearson Improved	2.5	Tracy	10/2/55	6.7	0.16
Ace	2.5	Tracy	10/2/55	6.7	0.16
Pearson Improved	2.5	Tracy	10/2/55	6.6	0.16
Pearson X L	2	San Jose	10/23/55	5.3	0.17
Pearson X L	2	Sacramento River Delta	10/23/55	5.8	0.16
Pearson X L	1.5	Tracy	10/22/55	5.0	0.16
Cal. Pak No 1	2	Tracy	10/22/55	4.7	0.17
Pearson Imp. B	2.5	Tracy	10/22/55	3.8	0.16
Pearson A 1	2	Tracy	10/22/55	4.6	0.17
John Moran	1.5–2.5	Tracy	10/22/55	3.6	0.15
Grand Pak	1.5–2.5	Tracy	10/22/55	5.0	0.17
Early Pak 7	1.25	Tracy	10/22/55	5.9	0.16
Peto Early Grown	1.5	Tracy	10/22/55	6.2	0.16
Peto Pride	1.5	Tracy	10/22/55	5.4	0.15
Pearson C	1.5–2	Tracy	10/22/55	6.0	0.17
Early Pak L	2–2.5	Tracy	10/22/55	4.7	0.16
Pearson S	1.25	Tracy	10/22/55	5.9	0.17
Ace	1.25	Tracy	10/22/55	6.3	0.16
Pearson Imp.	2	Tracy	10/22/55	5.2	0.16

[1] Water used as blank—0.00 Optical Density.

to apply to the readings later obtained with commercial pastes, purees, and juices.

If difficulty is encountered with filtration of the tomato serum, the use of Pectinase as described by Luh *et al (1)* may be advisable.

As a point of reference, it is perhaps of interest to note that an experienced taste panel detects flavor differences on reconstituted juice when the tomato serums of 2 samples vary by 0.15 OD. Samples whose serums are above 0.70 optical density are usually termed "objectionable." Samples which this laboratory found to have a serum OD of over 0.85 were from a lot of cans which were evaluated as "off-grade" by U.S.D.A. inspectors.

Description of containers. All cans used in this work were plain, common coke 603 x 700 cans, with inside enameled ends.

Effect of temperature on optical density of tomato serum. The correlation of the optical density with the degree of heat damage due to severe process conditions is shown on Table 2. The burned samples reported were obtained under adverse commercial conditions such as breakdown of closing machine, pumps, etc. during which abnormally extended high temperature holds were encountered.

The effect of inadequate cooling conditions on the OD of serum of tomato paste was checked as follows. Freshly manufactured 46% tomato paste was water cooled to a can center temperature of 135° F, and the cans were then placed in a

TABLE 2

Effect of severe process conditions on optical density of tomato serums

No.	Description of sample	% Solids	Optical density
1	Extended recirculation in final evaporating pan	46	0.85
2	Extended recirculation from filler bowl to preheater during closing machine stoppage	30	0.17
3	Pump breakdown between 2nd and 3rd effect on triple effect pan	30	0.34
4	Extended hold throughout line due to closing machine stoppage	26	0.13
5	Paste taken from line just prior to stoppage in Sample 4	26	0.02

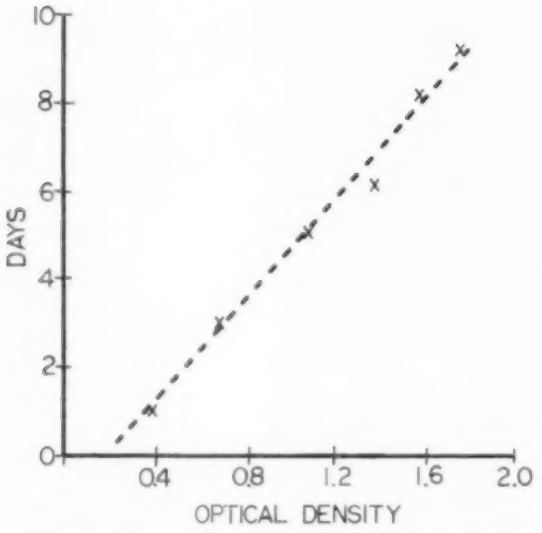

Figure 2. Increase in optical density of serum from 46% tomato paste stored at 130° F.

130° F oven. Samples were drawn over a period of 9 days for analysis. Results are shown in Figure 2.

The effect of storage temperature on OD of serum from tomato products was determined over a six months' period. Juice (8.8% solids) and paste (26% and 46% solids) were stored at different temperatures. The data in Figure 3 demonstrate that raising the storage temperature has a marked influence on rate of increase in OD of the serum. As the solids of the canned tomato product are increased, the influence of temperature becomes more pronounced.

Effect of optical density on expected shelf life of tomato paste. The shelf life of a pack of commercial 46% tomato paste with different OD stored at 70° F is shown in Figure 4. These differences were obtained as follows. The OD of 0.03 was obtained under the cannery's normal water cooling procedure. The 0.05 OD was obtained by bright stacking and air cooling

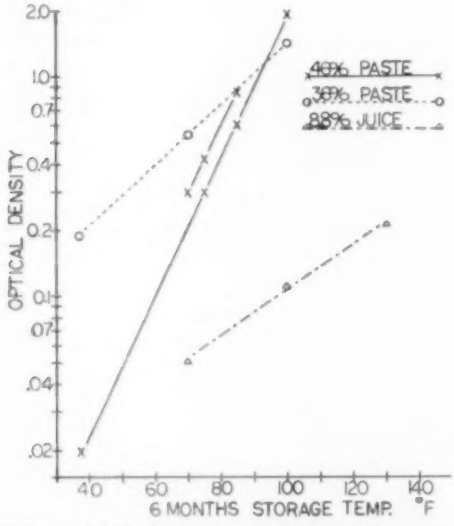

Figure 3. Optical density of serums from juice and paste after 6 months' storage at 37° F, 70° F, 85° F and 130° F. Data shows a straight line semi-logarithmic plot of a first order reaction.

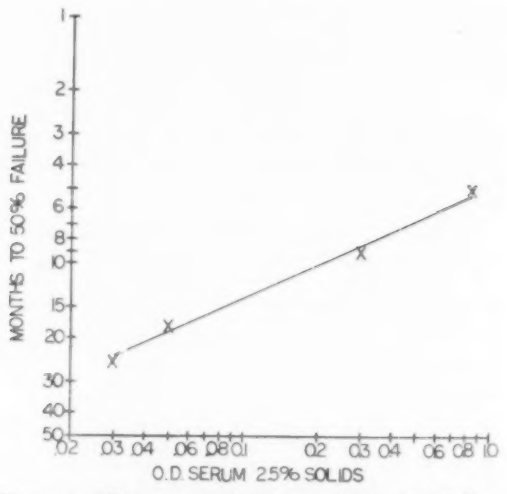

Figure 4. Effect of caramelization on time to 50% failure of 46% tomato paste in 603 x 700 Common Coke cans. Data is presented as a straight logarithmic plot. Lack of linearity of the data on rectangular coordinate, semilogarithmic, or reciprocal paper indicates that the kinetics are not presentable as a simple zero, first, or second order reaction. The rate of failure is therefore probably a complex of several interactions resulting in a fractional order reaction.

this same paste. The burning to 0.30 was obtained by casing the cans hot, and the OD of 0.85 was obtained by recirculating the paste in the filler bowl through a tubular heater. The plot of 50% failure of the cans against OD of the serum showed a startling decrease in service life as the OD at the time of canning was increased.

Effect of solids on shelf life of tomato pastes. The data reported in Figure 5 have been obtained over many years, from experiments in which the effect of solids was not the prime consideration. For this reason, factors other than solids content may have influenced service life. The data, however, show clearly that as solids of tomato products increase from that of

Figure 5. Effect of tomato solids on time to 50% failure of tomato products stored at 70° F in 603 x 700 Common Coke cans. Logarithmic plot is used. Lack of linearity of data on rectangular coordinate, semi-logarithmic, or reciprocal paper. As expected, the reaction is more complex than a single zero, first, or second order reaction.

puree of 18% solids to a paste of 40% solids, the expected shelf life of the product is reduced by over 5 fold when stored at room temperature.

DISCUSSION

The type of equipment used to measure light transmission of tomato serum varies from laboratory to laboratory. For this reason, color measurements have been reported in terms of OD measured with a Beckman Model B or Coleman Model 14, or Klett Readings. Use of different units makes it difficult to compare tomato serum color data obtained in different laboratories. As Luh *et al (1)* state, it is sometimes difficult to obtain clear filtrates. They report that incubation with Pectinol helps in obtaining clear filtrates.

To assist in making tomato serum color measurements and expressing results in terms independent of the instrument used, the following analytical procedure is suggested.

Mix the sample with sufficient water to give 2.75% solids. Centrifuge, then remove 20.0 ml of the supernatant liquid. To this add 2.0 ml of a freshly prepared 10% Pectinol suspension and incubate 90 min at 105° F. Then add about 0.2 g Supercel, mix, and filter through a fine filter paper. Measure light transmission of the clear filtrate at 420 mmc. Prepare a reagent blank, including the Pectinol, and measure its transmission. Correct the reading of the serum for this blank. Calibrate the equipment by making a series of standard potassium dichromate solutions and measuring their light transmission under the same conditions used for the tomato serum. Prepare a calibration curve or instrument constant to convert data for the tomato serum to a color index which is: the meg of potassium dichromate per ml of solution that has a transmission at 420 mmc equal to that of the clarified tomato serum (2.5% tomato solids) measured under the same conditions. Report results of color measurements in terms of this color index.

The results obtained here indicate that serious consideration should be given before increasing the solids of tomato paste in a commercial operation. It is now obvious that tomato paste of very high densities should be manufactured with the utmost care, and warehousing should be under the best of conditions. The method presented here will help those who use it to determine quickly how well they are succeeding in holding down process damage, and in evaluating their warehousing practices. The double effect on service life of high solids and heat damage to the product at any point, has resulted in pastes which, in test packs, had 50% failure in less than 3 months.

SUMMARY

The optical density of serum from tomato products has been found to be a useful measure of heat damage during production and storage. Raising the storage temperature or increasing the solids in the product accelerates darkening of the serum. Service life of the canned product decreases as optical density of the serum, per cent solids of the tomato product, or storage temperature is increased. The desirability of protecting high solids tomato paste from heat damage during production and storage has been demonstrated.

A method has been developed for measuring light transmission of tomato products and reporting results in terms of a color index that is independent of the equipment used to make the measurements.

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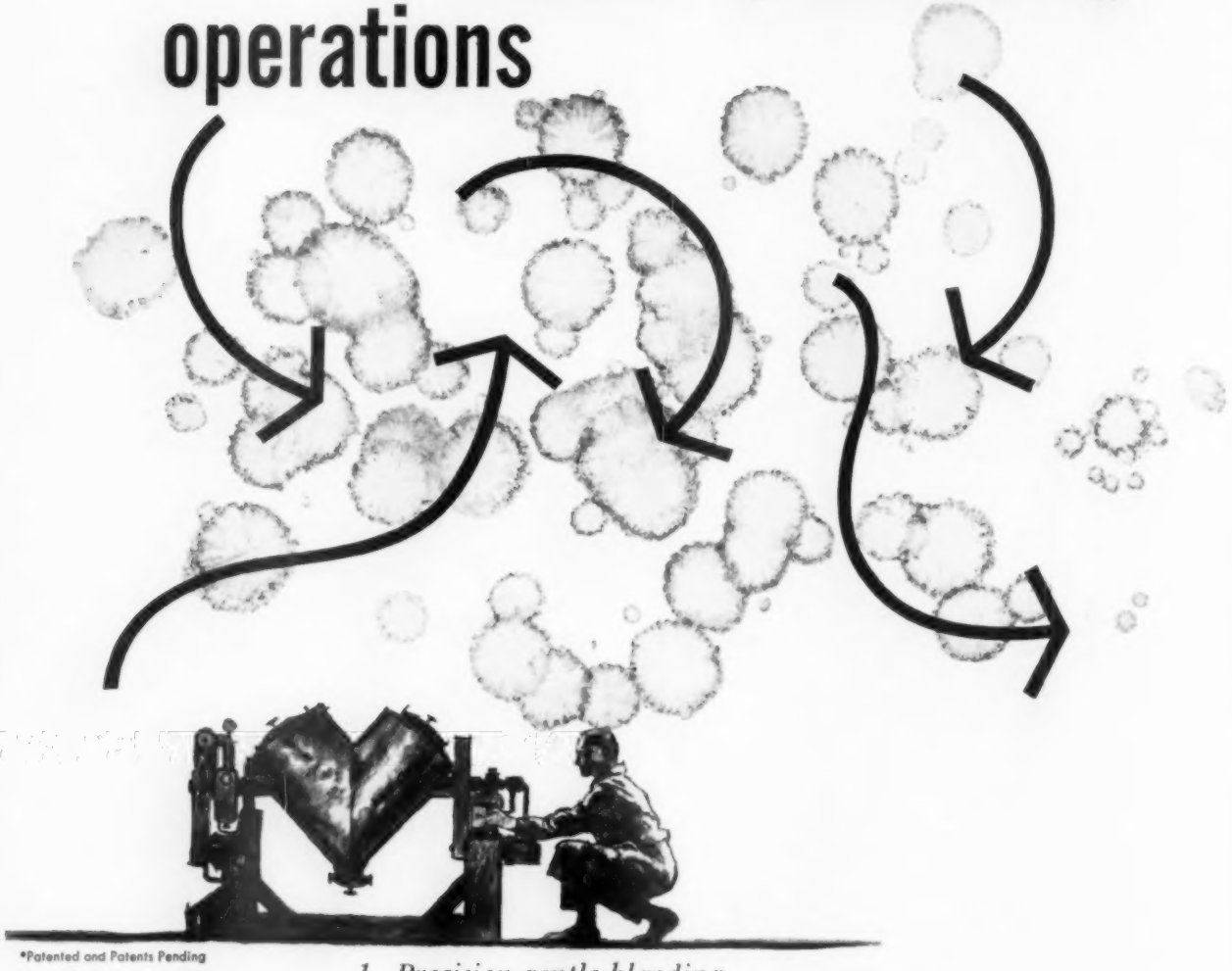

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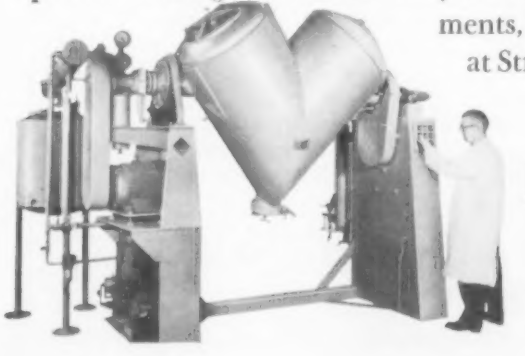

Patterson Kelley

Chemical and Process Equipment Division
116 Burson Street, East Stroudsburg, Pa.

(Continued from page 15 preceding Technical Section)

solids-acid ratio in the fresh fruit. The rate of change of pressure test prior to and at harvest was too low to provide reliable differences. Conversely, soluble solids-acid ratio changed rapidly in this same period and had a direct bearing on product quality. In addition the ratio was closely correlated with heat unit accumulation over the two seasons studied. Sauce color, flavor and grain improved as harvest was delayed to allow the fruit to tree-ripen. This was particularly noticeable if apples were processed into sauce directly after harvest. However, even when fruit was ripened to a common extent, as measured by pressure test or accumulated heat units, sauce from late-harvest apples was still noticeably better. Pressure test and calendar date of harvest were shown to be inferior indices of maturity, but heat unit accumulation and days after full bloom were observed to be about equally effective. The best prediction of apple maturity in 1958 based on results obtained in 1957 would have involved a compromise between the latter two indices. (See page 463).

Measurement — *Heat damage during production, storage of tomato products*

Optical density of tomato serum from concentrates as a measure of heat induced changes in product corrosivity. H. H. Hernandez and J. F. Feaster.

The optical density of serum from tomato products was found to be a useful measure of heat damage during production and storage. Raising storage temperature or increasing solids in the product accelerates darkening of the serum. Service life of the canned product decreases as optical density of the serum, per cent solids of the tomato product, or storage temperature is increased. The desirability of protecting high solids tomato paste from heat damage during production and storage was demonstrated. The method measures light transmission of tomato products and reports results in terms of a color index independent of the measuring equipment. (See page 468).

Dr. Roy Appointed Councilor-at-Large

President Hutchings announces the election of Dr. Wallace R. Roy, Vice-President, Minute Maid Corporation, Orlando, Florida, as Councilor-at-Large, to fill the vacancy created by the death of George F. Garnatz. He will serve in this capacity until the close of our 1961 Annual Meeting.

Dr. Roy received a Bachelor of Science degree in Chemistry from Clemson College, an M.S. from the University of Kentucky, and a Ph.D in Biochemistry from the University of Minnesota. He has for a number of years been prominent in IFT affairs, having held numerous Section offices and participated on IFT Committees.

Dr. Roy has been designated General Arrangements Chairman for the 1962 IFT Annual Meeting, to be held at Miami Beach, June 10–14.

PERSONNEL

Dr. H. E. Robinson, Chicago Section Chairman and vice president in charge of the research laboratories, engineering research and equipment development, Swift and Company, has assumed direction of a number of products-for-industry operations succeeding the recently retired E. A. Moss. Departments now under Dr. Robinson, in addition to general and engineering research, include adhesives, gelatin, stabilizers, soap and chemicals and the Technical Products Plant at Hammond, Ind. Dr. Robinson joined the Swift research staff in 1932, became assistant director of research in 1941, assistant director of laboratories in 1950, director of laboratories in 1953 and was elected vice president in 1959.

Dr. Imri J. Hutchings, IFT President and manager of research, H. J. Heinz Company, has been appointed to the Science and Advisory Committee of Duquesne University, at Pittsburgh, Penna. Dr. Hutchings will serve on the committee with Dr. John C. Warner, president, Carnegie Institute of Technology; Dr. Blaine B. Wescott, executive vice president, Gulf Research and Development Company; and E. J. Hanley, president, Allegheny Ludlum Steel Corporation. Function of the committee is to assist the faculty of the university in the continuing development of the physical and natural sciences within the school's academic framework.

I. J. Hutchings

Dr. Austin C. Wagenknecht, first recipient of a Gerber post-doctorate fellowship administered by the IFT, has been employed by the General Mills, Inc., Central Research Laboratories, Minneapolis, Minnesota as Principal Scientist in charge of lipid research in the Fundamental Food Research Department. He attended the University of Wisconsin where he received a bachelor's degree in chemistry, and his master's and doctor's degrees in biochemistry, and studied at the University of Illinois during the effective period of his Fellowship. Dr. Wagenknecht joins the Laboratories from the New York State Agricultural Experiment Station, Cornell University, Geneva, New York, where he held the position of Associate Professor of Biochemistry in the Department of Food Science and Technology.

EQUIPMENT

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ABSTRACTS

General Foods Research Center
Selected by H. A. Campbell

ANALYTICAL METHODS

Separation of some terpenoid compounds by gas-liquid chromatography.

RUDLOFF, E. *Can. J. Chem. 38*, 631–40 (1960).

A study has been made of the degree of sepn. of mixts. of some terpene hydrocarbons, some of their oxygenated derivs., 2 sesquiterpene alcohols, and 3 monophenols on a variety of columns. Temperature and sample size affected the degree of sepn. and the solid support Chromosorb W, a calcined diatomaceous earth, was found to combine the advantages of Celite and C-22 firebrick without causing decompn. of the sample at higher temps. When Craig polyesters were used as liquid phases, sepns. equal to those obtained on polyethylene glycol were realized with the added advantage that these produced columns which are stable at 190 to 220° C. Consequently, sesquiterpene alcohols and monophenols were also sepd. successfully.

Analysis and composition of oil of lemon by gas-liquid chromatography.

BERNHARD, R. A. (Univ. of California, Davis). *J. Chromatography 3*, 471–6 (1960).

Cold-pressed lemon oil was examd. by means of gas-liquid chromatography and a number of the constituents present was tentatively identified by means of corrected retention volume correlation employing 2 stationary liquid phases. The components identified by these procedures were: α-pinene, β-pinene, myrcene, D-limonene, n-heptanal, 1,8-cineole, γ-terpinene, n-hexanol, n-octanal, methyl heptenone, n-nonanal, linalool, n-decanal, citronellyl acetate, citronellal, n-undecanal, tetrahydrogeraniol, decyl acetate, geraniol, α-terpineol, citronellol, neral, geranyl acetate, citral, and D-carvone.

Determination of free fatty acids in fat.

HORNSTEIN, I., ET AL. (Eastern Utilization Research and Devel. Div., Beltsville, Md.). *Anal. Chem. 32*, 540–2 (1960).

Naturally occurring free fatty acids may be detd. in the presence of large amts. of unsaponified fat by adsorbing the free fatty acids on a strong anion base exchange resin, washing the resin free of fat with petroleum ether, and converting the free fatty acids directly on the resin to their methyl esters with anhydrous methanol-hydrochloric acid. The nature and concn. of the fatty acids are detd. by gas chromatography.

Rapid combustion and determination of residues of chlorinated pesticides using a modified Schoniger method.

LISK, D. J. (Cornell Univ., Ithaca, N. Y.). *J. Agr. Food Chem. 8*, 119–21 (1960).

A method is described for the rapid combustion and detn. of residues of chlorinated pesticides. Org. solvent exts. of plant samples are evapd. in a cone of cellulose acetate, which is burned in an oxygen-filled (Schoniger) flask and the HCl gas is absorbed in dil. NaOH. The spectrophotometric detn. of chloride is based on its displacement of thiocyanate in the presence of ferric ion. The time required for combustion and detn. of chloride is about 20 min./sample.

BIOLOGICAL SCIENCES

Biochemistry

Immunoelectrophoretic studies on some modified proteins. I. Human serum albumin. (In French).

STEINBUCH, M., FINE, J. M. AND BATTISTINI, A. *Clin. Chim. Acta 5*, 345–8 (1960).

Modified human albumins are studied by zone electrophoresis on paper, starch gel, and agar, by immunoelectrophoretic analysis and by the agar diffusion technique. Blocking of amino groups alone or combined with the introduction of new carboxyl groups results in acceleration of the electrophoretic mobility at pH 8.6 and in decrease of affinity for the usual acid dyes. The immunological reactivity towards horse or rabbit anti-human sera is not impaired by the blocking of the free amino groups. Blocking of the free carboxyl groups of albumin results in a diminution of the electrophoretic mobility and an increase in staining. Esterification of the carboxyl groups suppresses the immunological activity, in proportion to the degree of esterification.

Changes in the reactivity of disulfide bonds in bovine serum albumin on denaturation.

ALEXANDER, P. AND HAMILTON, L. D. G. (Roy. Cancer Hosp., London, Engl.). *Arch. Biochem. Biophys. 88*, 128–35 (1960).

Earlier observations showing that in native bovine serum albumin none of the disulfide bonds can be reduced at pH 5.4 have been confirmed. They are also resistant to oxidation by dil. peracetic acid, proving that failure to react must be ascribed to steric factors and cannot be attributed to alterations in the redox potential which prevents reduction. Denaturation was shown to occur in discrete steps, and 3 physicochemically distinct products have been isolated. The first step leads to the revelation of 25% of the disulfide bonds without changing the soly. The second stage is characterized by 40–50% reactive disulfide bonds and failure to dissolve in water, although remaining soluble in solns. of high ionic strength as large aggregates.

Oxidation of sulfihydryl groups in denatured egg albumin.

TSEN, C. C. AND TAPPEL, A. L. (Univ. of California, Davis). *Can. J. Biochem. Physiol. 38*, 539–44 (1960).

The oxidation of dry denatured egg albumin and dry myosin by molecular oxygen indicates that the direct oxidation of dry proteins contg. free sulfhydryl groups is a general reaction. Cupric ion was the most effective catalyst for the oxidation of denatured egg albumin in soln. The rate of the oxidation was measured as a function of cupric ion concn., pH, and temp. Since the catalytic oxidations of denatured egg albumin, glutathione, and cysteine have very similar activation energies, the mechanism of copper-catalyzed sulfhydryl oxidation may be the same for the 3 compds. Under the conditions studied, only sulfhydryl groups of denatured egg albumin were oxidized. The oxidation of these groups proceeded beyond the disulfide stage.

Enzymatically active products of trypsin autolysis.

WAINFAN, E. AND HESS, G. P. (Cornell Univ., Ithaca, N. Y.). *J. Am. Chem. Soc. 82*, 2069–72 (1960).

The autolysis of trypsin under appropriate conditions has been found to result in enzyme molecules which dialyze through 18/32 Visking cellophane membranes and whose dialysis rate through the larger pore 20/32 membranes is more rapid than that of trypsin.

Estrogen-enzyme interactions: Inhibition and protection of kynurenine transaminase by the sulfate esters of diethylstilbestrol, estradiol, and estrone.

MASON, M. AND GULLEKSON, E. H. (Univ. of Michigan, Ann Arbor). *J. Biol. Chem. 235*, 1312–16 (1960).

In a study of the effects of various steroids on the kynurenine transaminase system of rat kidney, estradiol disulfate and diethylstilbestrol disulfate were found to inhibit at levels as low as 5×10^{-7} *M*. Estrone sulfate and pregnanediol glucuronide inhibited only at much higher concns. A number of non-anionic steroids, including estradiol, diethylstilbestrol, and estrone, were not inhibitory even in satd. solns. The estrogen sulfates also interact with the kynurenine apotransaminase in a way that prevents the inactivation accompanying incubation.

Microbiology

Survival of Salmonella typhimurium, Staphylococcus aureus, and Streptococcus faecalis frozen in simplified food substrates.

WOODBURN, M. J. AND STRONG, D. H. (Univ. of Wisconsin, Madison). *Appl. Microbiol. 8*, 109–13 (1960).

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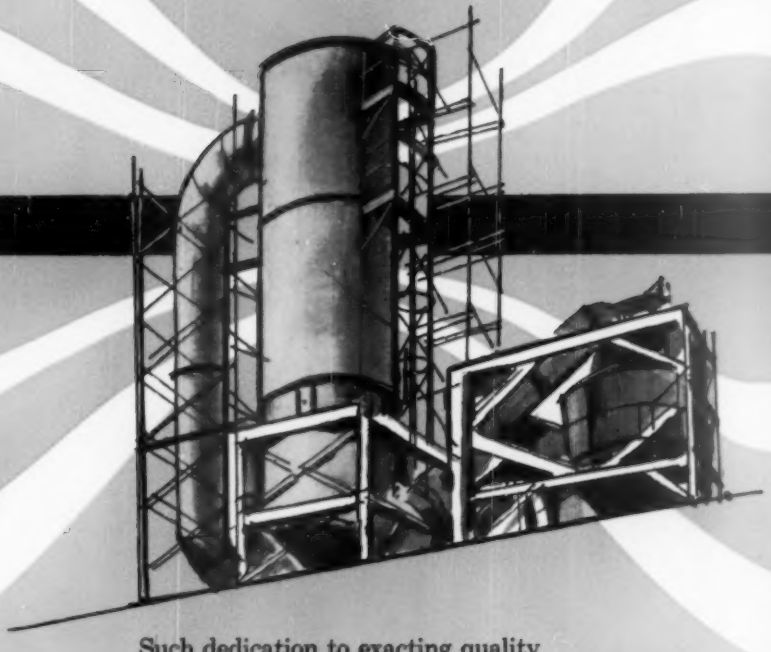

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The organisms used for inocula were *Salmonella typhimurium* 196, *Staphylococcus aureus* 84, and *Streptococcus faecalis* R26. Periods of frozen storage extended through 10 wks. at −11, −21, and −30° C. The addn. of the rice flour, corn sirup, or egg white to the phosphate buffer resulted in the maintenance of increased numbers of viable cells at each temp. and time under the exptl. conditions. The extent of this effect for each ingredient differed with the organism, time and temp. These differences were most marked in the recovery of viable cells from the sodium alginate medium.

Peptides and bacterial growth. IX. Release of double inhibitions with single peptides.

KIHARA, H. AND SNELL, E. E. (Univ. of California, Berkeley). *J. Biol. Chem. 235*, 1415–18 (1960).

High concns. of L-alanine, L-leucine and β-2-thienyl-L-alanine inhibit growth of *Leuconostoc mesenteroides* P-60 by restricting utilization, resp., of the essential amino acids glycine, L-isoleucine, and L-phenylalanine. In the presence of any one of these inhibitors, growth is restored by addn. of large amts. of the proper limiting amino acid, or by comparatively small amts. of utilizable peptides of that amino acid. When 2 of the inhibitors, e.g. alanine and leucine, are added, growth is restored by large amts. of glycine plus isoleucine, by small amts. of a mixt. of dipeptides supplying these 2 amino acids, or with equal efficiency by a single dipeptide, glycyl-L-isoleucine, that supplies both amino acids.

Nutrition

Effects of cholesterol and other substances on essential fatty acid deficiencies.

GAMBAL, D. AND QUACKENBUSH, F. S. (Purdue Univ., Lafayette, Ind.). *J. Nutrition 70*, 497–501 (1960).

Cholesterol and sodium glycocholate accelerated EFA-deficiency symptoms using diets contg. 3% of satd. fat. Both substances retarded growth. Cholic acid did not accelerate EFA-deficiency symptoms, but retarded growth. When cholesterol was fed with either sodium glycocholate or cholic acid, the lipid content of the liver increased several fold. However, sodium glycocholate was the more effective of the 2 supplements, with cholesterol, to increase liver lipids and accelerate EFA-deficiency symptoms.

Diet as source of serum cholesterol in man.

TAYLOR, C. B., ET AL. (Presbyterian-St. Luke's Hosp., Chicago, Ill.). *Proc. Soc. Exptl. Biol. Med. 103*, 768–72 (1960).

Normally active healthy adult volunteers ingested cholesterol-rich diets labelled with 4-C-14-cholesterol. The diets supplied enough cholesterol to suppress hepatic cholesterol synthesis, if the liver is as responsive to dietary cholesterol in man as in lower animals. Radioactivity of serum cholesterol permitted direct calcn. of amt. of serum cholesterol coming from the diet, which would equal the amt. that would come from the liver if the diet supplied none. This amt. was found to be 24 to 31%. Presumably, then, at least ¾ of serum cholesterol of these human subjects was derived from cholesterol synthesis in extrahepatic tissues.

Infant-feeding practices and blood cholesterol levels.

POMERANZE, J. (New York Med. Coll., New York City). *Am. J. Clin. Nutrition 8*, 340–3 (1960).

Serum cholesterol levels of infants fed an evaporated milk formula were higher than those in infants fed a prepd. milk formula contg. a high level of unsatd. fatty acids. Serum cholesterol levels of infants fed breast milk were considerably higher than would be expected since breast milk is relatively high in unsatd. fatty acids. This may result from the relatively high caloric and high satd. fat intake by American mothers. The author suggests that if dietary control is to be used to prevent atherosclerosis later in life we should give consideration to modification of the infant diet with higher levels of unsatd. fatty acids.

Individual metabolic patterns. I. Amino acid excretion studies in 700 children.

BERRY, H. K. (Univ. of Cincinnati, Ohio). *Metabolism: Clin. and Exptl. 9*, 363–72 (1960).

Significant differences were found in the amino acid excretion patterns detd. by paper chromatography among 687 normal children and children with different diseases varying in age from 1 day to 16 years. Infants excrete greater amts. of amino acids than older children. Glycine, alanine, glutamine, histidine, proline and hydroxyproline are the more prominent amino acid constituents of urine during the early months of life. Proline and hydroxyproline are excreted in only trace amts. after 6 months.

Effects of amino acids on the excretion of various proteins by the rat.

HARDY, R. W. F., ET AL. (Univ. of Wisconsin, Madison). *J. Nutrition 70*, 438–46 (1960).

Rats were given large doses of single amino acids by stomach tube, and measurements were made of urinary protein, urea and α-amino nitrogen. Most of the amino acids increased protein excretion roughly in proportion to the increased urea production. DL-Serine, D-serine and L-cysteine·HCl produced excessive elevations in protein excretion, whereas glycine and alanine depressed it.

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SINCE 1853

Role of rate of ingestion of diet on regulation of intermediary metabolism ("Meal Eating" vs. "Nibbling").

COHN, C. AND JOSEPH, D. (Michael Reese Hosp., Chicago, Ill.). *Metabolism: Clin. and Exptl. 9*, 492–500 (1960).

The findings are interpreted to indicate (a) that flooding of the organism with calories by the consumption of full meals is assocd. with a more economical use of calories, leading to obesity; (b) that meal eating differentially affects the traffic over alternate metabolic pathways; and (c) that the rate of TSH release by the pituitary may be decreased by meal eating. The observed phenomena may be mediated through alterations in the enzymatic machinery of the body secondary to different rates of influx of calories.

Nutrition and the formation of urinary calculi.

BOYCE, W. H. *Borden's Rev. 21*, 27–36 (1960).

The precise mechanisms by which uromucoid is transformed into matrix and by which it accretes org. or inorg. crystals to become an organized urinary stone are presently unknown. Current evidence is overwhelmingly indicative of a dietary deficiency as the important nutritional aspect of spontaneous human calculi. The deficiency may eventually prove to be relative and multiple rather than absolute and singular. On the other hand, there is no evidence that dietary surfeit without nutritional deficiency is ever an important factor in human stone formation.

Toxicology

Toxicity studies on dibutyl- and diamylhydroquinone.

WILSON, R. H. AND POLEY, G. W. (Western Regional Research Lab., Albany, Calif.). *Proc. Soc. Exptl. Biol. Med. 104*, 29–31 (1960).

Two hydroquinone derivs., 2,5-di-tert-butylhydroquinone and 2,5-di-tert-amylhydroquinone, have been suggested as antioxidants to protect carotene of dehydrated alfalfa meal. Neither compd. modified growth, appearance, organ wts. or histological structure of rats eating diets contg. as much as 0.2% of antioxidants for 500 days. No symptoms of intoxication developed in exploratory tests on rats when single doses of 1500 mg./kg. of compds. were given by stomach tube. These materials did not sensitize the skin of guinea pigs but did produce a reversible lesion on rabbit and guinea pig skin when applied daily to the skin as 10% solns. in cottonseed oil.

Studies on a modified fat.

COLEMAN, R. D. (Univ. of Southern California). *Dissertation Abstr. 20*, 3940–1 (1960).

In studies on rats, growth was markedly retarded in animals fed acetostearin

as the sole fat at relatively high levels. It was considerably improved in animals supplemented with linoleate and markedly improved when cottonseed oil was fed with the acetostearin. Mortality was high in groups fed acetostearin alone, but decreased when acetostearin diets were supplemented with a source of essential fatty acids. Cholesterol analysis on tissues of rats fed acetostearin as the sole fat revealed a decrease in the plasma cholesterol level and an increase in the cholesterol in the liver.

FOOD AND FOOD TECHNOLOGY

Baking and Bakery Products

A role for thiol and disulfide groups in the rheological properties of dough made from wheaten flour.

FRATER, R., ET AL. (Univ. of Melbourne, Victoria, Australia). *Nature 186*, 451–4 (1960).

Evidence is presented to support the hypothesis that strength and elasticity of doughs are related to thiol groups, intermolecular disulfide bonds and the rate at which they interchange. The action of improvers is also explained in terms of disulfide interchange.

PHYSICAL METHODS

Ultra-violet spectra of water solutions.

GOLDING, R. M. (Dept. of Scientific and Industrial Research, Wellington, New Zealand). *Nature 186*, 308 (1960).

The spectrum of pure water at room temp. was found to be relatively weak and to have three main peaks at 1880, 1890 and 1908 A.

INDUSTRY NEWS

Freeze Drying and Other Dehydration Methods of Food Preservation will be the theme of a military-industry conference sponsored by the R&D Associates, set for September 20–21, 1960, at the Saddle & Sirloin Clubrooms, Stock Yard Inn, Chicago. In charge of the meeting are Dr. J. J. Jackson of American Can Company, Vice President in Charge of Activities, and Mr. George A. Crapple, Wilson & Company, Vice Chairman of Activities.

The program the first day, September 20th, will cover the latest developments in food dehydration, including economic factors and related packaging problems. Speakers will be representatives of food companies now in the dehydration field, government and university research institutions at work on specific projects, and staff members of the QM Food and Con-

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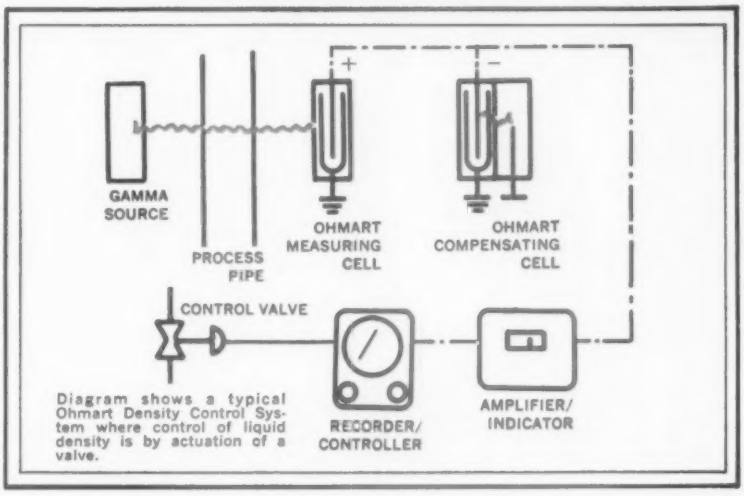

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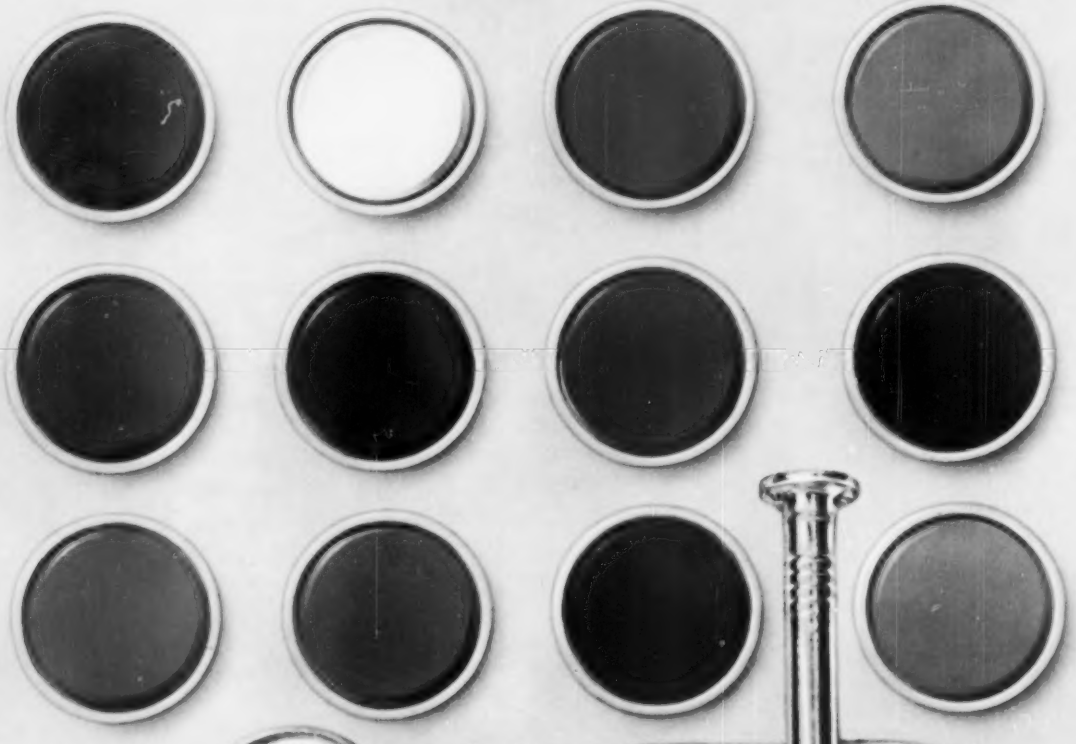

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tainer Institute for the Armed Forces. Subjects scheduled to date are: *Foam-Mat Drying; Progress and Plans in the Development of Pre-Cooked Dehydrated Sweet Potato Flakes; Practical Principles of Industrial Freeze-Drying; Perfect Closure on Flexible Packaging; and Developing "Locked-In" Citrus Oil Flavors, Dehydrated Citrus Ades and Dehydrated Citrus Peel.*

Wednesday, September 21, will be devoted to a symposium of freeze-drying and other types of dehydration equipment. Panel participants will be representatives of leading equipment companies in the United States as well as from foreign countries. It is planned to display models of the various kinds of equipment to be discussed.

This will be an open meeting. A cordial invitation to attend is extended all personnel of firms in the food, packaging, and related fields who wish to keep alert to developments in the expanding market for dehydrated foods. Certainly any company considering the production or use of freeze-dried or otherwise dehydrated items, or in manufacturing equipment and supplies, should be represented. Write to Colonel Rohland A. Isker, ret'd, Executive Secretary, Research and Development Associates, Food and Container Institute, Inc., 1819 W. Pershing Road, Chicago 9, Illinois, for reservations and further details.

The Food and Allied Industries Committee of the American Society for Quality Control, organized to achieve National Divisional Status during the 1960–61 fiscal year, will be concerned with the application of statistical quality control to food and allied processes. It will provide a medium for exchanging statistical quality control information in these industries and to consider SQC problems unique to this field. Quality control personnel in the food and allied industries should contact the following: William A. Golomski, Chairman, c/o H. W. Mayer and Sons, 6811 S. Ashland Avenue, Chicago 36, Illinois; Mae-Goodwin Tarver, Secretary-Treasurer, c/o Continental Can Company, Inc., 1350 W. 76th Street, Chicago 20, Illinois.

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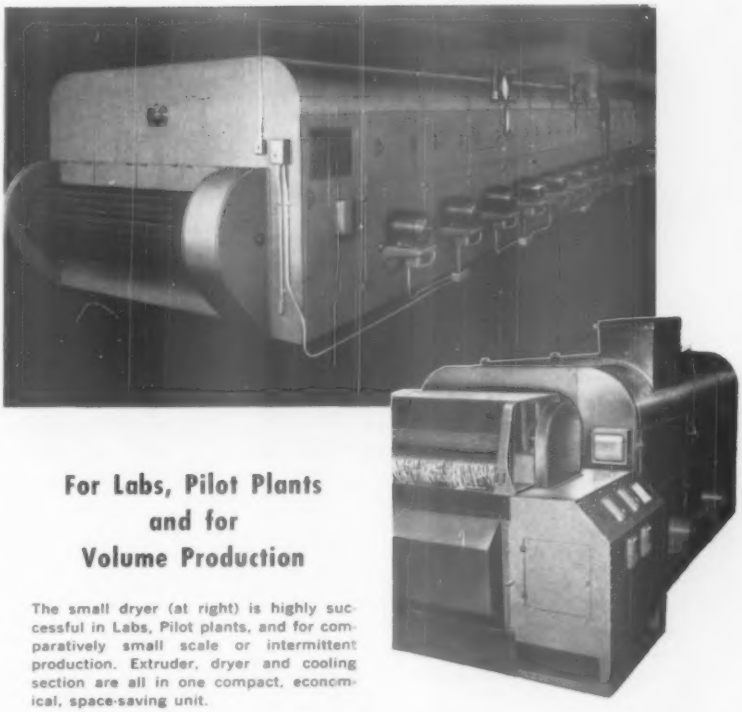

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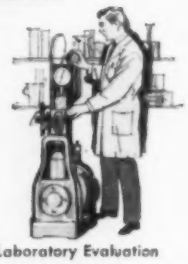

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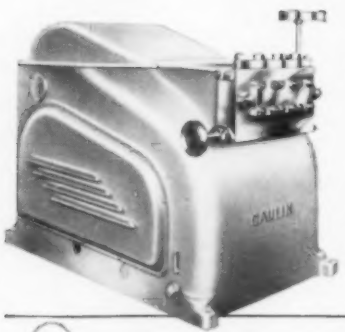

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WANTED: DAIRY TECHNOLOGIST-Conduct Research and Development work on dairy products. Knowledge of vitamin fortification of dairy products and coloring agents for butter (or cheese), washing compounds for bottles. Experience starter cultures. Will be Tech. Information Source for questions regarding dairy products. Ph.D. in Dairy Tecn. Prefer Eastern U.S. resident. 5 yrs. industrial experience in dairy work. REPLY BOX 806, Institute of Food Technologists, 176 W. Adams St., Chicago 3, Ill.

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FOOD ENGINEER - National food company desires man with B.S. in Food Technology or Chemical Engineering for opening in food product development. Work includes product formulation and process engineering. Experience in food industry desirable but not mandatory. Salary commensurate with training and experience. Write to Dr. G. Reed, Director of Research, Red Star Yeast and Products Co., P.O. Box 737, Milwaukee 1, Wisconsin.

WANTED: SPICE & SEASONING TECHNOLOGIST-Challenging opportunity for Product Development. Expanding national spice and seasoning firm desires graduate chemist 3-10 years experience. Salary commensurate with training and experience. Many benefits. Company develops and manufactures ingredients for food processors. Send resumé to:

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Prefer Food Technologist or related with Ph.D. and 2-3 years pertinent experience or M.S., B.S. with 4-8 years pertinent experience. Knowledge of statistical methods desirable. B.S. Food Technologists with limited experience are invited to inquire about other openings in product development.

Liberal employee benefits and relocation allowances. Please send resumé and salary requirements to:

Manager, Technical Employment
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RESEARCH BIOCHEMIST — Position requires a strong background in chemistry, bio-chemistry and/or Food Technology. Assignments will involve research on applications for company products, primarily enzymes, and technical service. Educational requirements are B.S. degree; M.S. desirable but not necessary. Experience: 0-3 years. Miles Chemical Company, Clifton, New Jersey.

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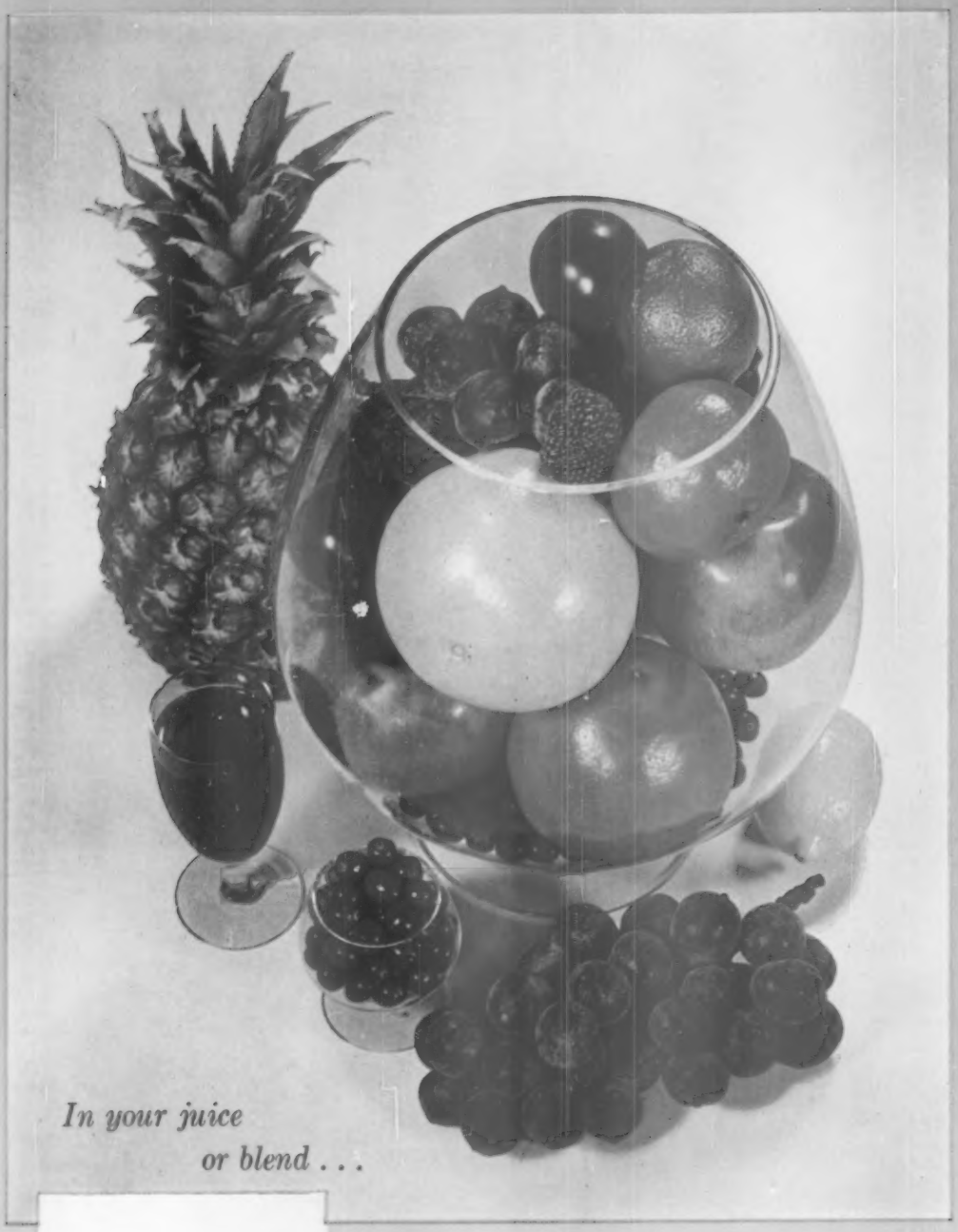

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